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VOLUME XII AUGUST 14, 1923 NUMBER 17

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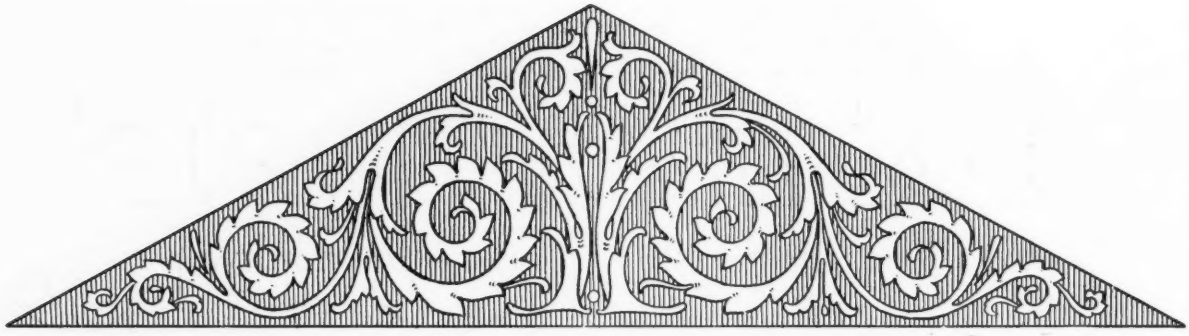

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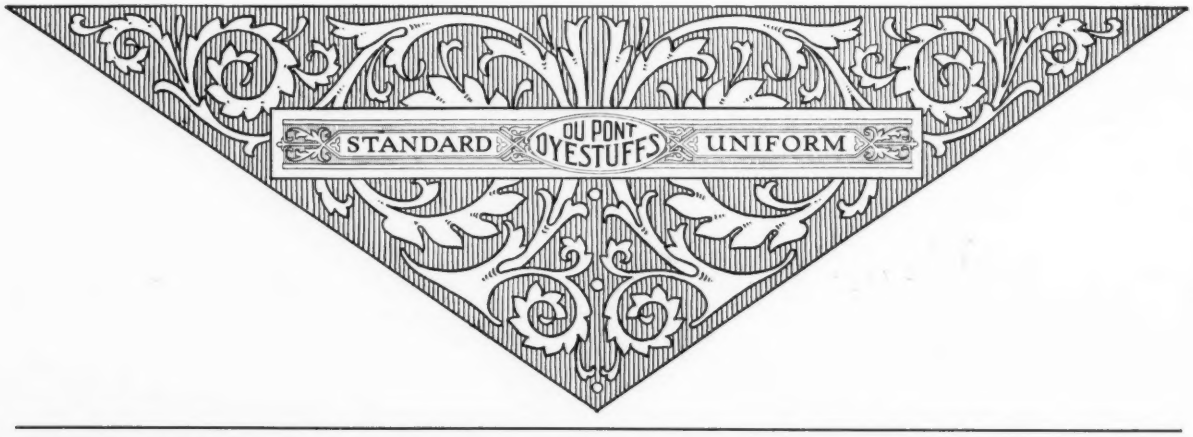

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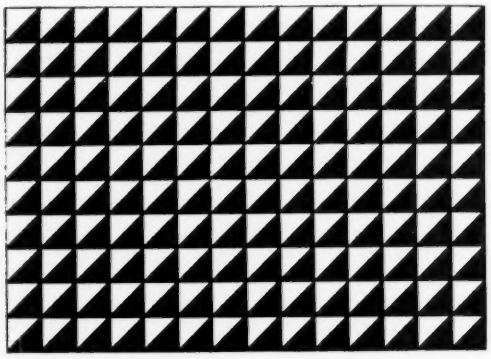

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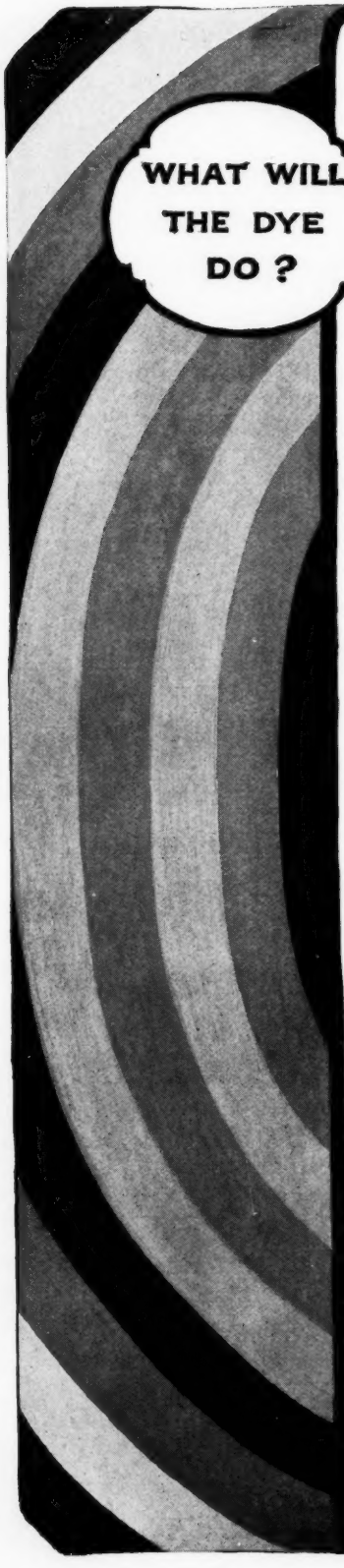

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VOLUME XII NEW YORK, AUGUST 13, 1923 NUMBER 17

Indigo Dyeing in China

Use of the Fermentation Vat—Where Natural Indigo Is Preferred—Manufacture of "Nesell"—The Piece Goods Shops—Solid Shades and Resist Dyeing—Chinese Dye Vats and Methods of Procedure—Starting a New Dye Vat—Manipulation of the Cloth—Drying and Finishing—Resist Styles—Results

By WOO DONG CHI

FOR textiles, blue is the most prevalent color in China, and fully 90 per cent of it is produced with Indigo. China consumes fully three times as much Indigo as Japan, her nearest rival. In 1922 China imported and used in the neighborhood of thirty million pounds of Indigo 20 per cent paste. Given such an important consumption we think our American friends will be interested in a brief description of how, where, and to what, it is applied.

The bulk of the Indigo consumed is dyed on cotton cloth. Small quantities are applied to silk and some to wool, the latter only for making the so-called Tientsin carpets.

No matter to what material it is applied, the dyehouse, apparatus and method of dyeing are essentially the same. Except in parts of Chili Province and in Manchuria, which use zinc lime, China is still using the fermentation vat.

There are three essentials in this vat—the coloring matter, a decomposing material to feed the Indigo ferment, and an alkalining agent.

Use of the Fermentation Vat

There are three general methods for carrying on the fermentation: (1) In parts of Kiangsu and Chekiang Province, which we may call the Shanghai district, lime is the alkalining agent. The rice hull mash left from making the native wine, samshu, is added as food for the bacteria. (2) In most of the provinces bordering on the Yangtze River lime and soda ash together are used for alkalining. The bacteria are fed by small additions of low-grade native Indigo containing much organic impurity, bran, samshu itself, and similar materials. (3) In districts for which Canton is the center caustic soda is the alkalining agent, while the ferment is fed with bran, etc., as in the second method.

The Shanghai method seems to be the best. The vats are controlled more easily and seldom get out of order, while the exact opposite is true of the others, especially the Yangtsze method.

Since China is still undeveloped industrially, it is to be expected that her most important branch of dyeing remains in the nature of a home industry. The fairly large modern spinning and weaving mills operated by foreign and Chinese capital are confining themselves very much to the manufacture of gray or bleached calico. Most of them have no dyehouses at all and those that have such departments do not dye Indigo. The dyehouses, apparatus, and methods of procedure for Indigo dyeing, therefore, still follow the Chinese practice as evolved during centuries, and are not influenced by modern methods. Even so, the results produced are satisfactory to the consumer and the costs are low. Modern dyehouses and large-scale dyeing operations will not appear for many years, due to the method of cotton cloth marketing and to the transportation and taxation difficulties which beset our vast country.

Much foreign woven calico, bleached and gray, is sold throughout the country, but the bulk of the Indigo is applied to the domestic cloth—of which the larger part is still woven in the home on hand looms.

Cotton is grown in many provinces in China, and so in very early times crude spinninng and weaving industries sprang up around the cotton fields. Probably not long afterward the art of applying natural Indigo was developed. Until the production of synthetic Indigo was a commercial possibility China was practically self-supporting in Indigo cultivation.

Indigo could be grown in many cases near the cotton fields so that transportation was not a factor. Of course, artificial Indigo had nearly killed this industry at the time the World War shut off supplies, but even in 1913 the natural variety was being produced in a few districts. From 1914 on cultivation increased rapidly, spreading from the old strongholds to nearly every province.

Where Natural Indigo Is Preferred

Since the hard times of 1920-21 natural Indigo production has been on the down grade due to return of the synthetic variety to pre-war conditions as to quantity and price. However, it will be many years, and probably not in this generation, before cultivation is entirely extinct. The prejudices of old dyers and wearers of textiles for the natural variety—a prejudice strong in many lands—was thoroughly broken by the sellers of the artificial product before 1914. It is only where transportation difficulties and distances from importing centers are very great that natural Indigo will hold its own. To mention only two such districts in central and western China, where these conditions remain, there are certain parts of Kiangsi Province and the Province of Szechuan.

A few other facts might be mentioned before leaving this phase of the subject. Sales of natural Indigo in Kiangsi Province, one of the biggest producing centers in the Yangtze Valley, in 1919 were about three million Mexican dollars.

Natural Indigo appears on the market in China as a thick paste packed in wooden tubs, holding from 135 to 200 pounds net. The best variety contains not over 3 per cent indigotin, and about 20 per cent solids. The poorest quality that it is profitable to ship, say 100 miles, contains around 1½ per cent indigotin and 30 to 35 per cent of solids.

Each tub of paste has to be sampled and valued by the prospective purchaser. One method of test is to spread some of the paste on a piece of unsized paper, where the shade and intensity of the blue stain produced can be judged.

The points we have tried to bring out so far are the fact that Indigo is the most important dyestuff used in China; that the amount consumed is very large; that most of it is applied to cotton in the form of calico piece goods either of foreign or domestic manufacture, of which the latter predominates; and that the dyeing is still essentially a home industry, using the same type of fermentation vat as was developed in this country when the present Western nations were not interested in dyeing spelled with a Y.

Growth of Great Home "Nesell" Industry

We have said that Indigo dyeing here is a home industry which first sprang up around the cotton fields. This home industry may still be seen to-day in countless places, some of them within easy reach of the foreign settlements in the treaty ports. The cotton is grown, picked, ginned, and spun on hand wheels by each family. The warps are "laid" on frames set up beside the fields or at one side of a village street. They are starched by hand with a brush and bean flour. Finally, they are rolled onto the "beam" and put into the hand loom, where the women and children of the family weave it into cloth.

The usual "fruit of the loom" is a coarse calico some 15 inches wide and 12 to 15 feet in length. A translation of the Chinese name for such cloth is "nesell."

As time went on this home textile industry grew and tended to specialize. This was especially true of the dyeing, due to the skill necessary to start and control the dye vats. Gradually some men specialized entirely on dyeing, and their dyehouses grew to large proportions, having as many as four hundred vats and one hundred and fifty to two hundred workmen. To-day there are many villages, and even cities, famous for Indigo dyeing alone, such as Shasi, on the Yangtze River in Huper Province, and Hingning, in the south, near Canton.

Another factor in the cotton cloth weaving and dyeing industry is the cloth merchant, or "piece goods shop" as it is called. In even the smallest villages such shops are found. They act as the middleman between producer and consumer. Such a shop carries all sorts of stuffs in demand as clothing materials, and in the last few decades this has included such material woven, dyed and finished abroad. Previous to this, however, they handled mainly native products, and it is with the native-made cloth that the bulk of their business is to-day. For this reason, it might be remarked in passing, the import and sale of foreign-made piece goods is not a reliable index to the volume of the dye business of China during any given period, since much of the imported material is not dyed here.

The Piece Goods Shop

The piece goods shops are ready to buy the native-made piece goods and distribute them as demand arises. They keep most of such stock in the gray and have it dyed or bleached to the order of each customer. Of course, there are certain standard shades of blue of which a small stock can be kept for each season. In general, medium and deep shades of blue are worn in fall and winter, and light sky blues during the spring and summer.

Some piece goods shops contract with the villagers for their supply of "nesell." Some piece goods shops have their own dyehouses, and some send their goods to commission dyehouses.

In the sale of Indigo it is thus often of more importance to convince the piece goods shops of the excellence of a particular brand than to convince the dyehouse owner himself. Sometimes the piece goods shop finances the dyehouse in the purchase of Indigo—which is its chief expense.

The method of marketing cotton piece goods, then, has had a tendency to create establishments which do not produce cloth and which specialize only on the dyeing and finishing of it.

Two Styles of Dyeing

At this point we may state that there are two gen-

eral "styles" of dyeing: (1) The plain solid dyeing in all depths of shade, from the lightest blue, on bleached pieces, to the deepest navy; and (2) the method of dyeing where a resist is applied to leave a white pattern.

The kind of vat (fermentation or otherwise) is the same in both cases, but the concentration of Indigo is greater in the second. The method of handling the cloth is varied also. Probably most of the resist dyeing is done in dyehouses rather than in the home, because of the greater dyeing skill required and also the greater amount of space necessary for applying the resist. Sometimes one dyehouse will dye both plain and resist "styles," but more often each style is dyed in a separate place.

The Indigo dyehouse of China differs greatly from a place of the same name in the United States. The dye vats are housed in one small room or group of rooms which often show how the establishment has grown from small beginnings. The floor is dirt or brick. The walls are low and the roof is tile. In many cases the only light comes from the doors, of which there may be two or more. Water is obtained from a well in or near the building, or from a stagnant pool, stream or canal near by.

The owner or foreman dyer lives in, or adjacent to, the dyehouse. The workmen also live there or near by. Accordingly, there are always many children and domestic fowl and animals in and around the building.

The principal part of the dyehouse is, of course, the dye vats. They are arranged in groups in the middle of the room or in rows along the walls. Sometimes both arrangements appear in one room. If placed in groups, they are in a double line of four, six, eight or twelve.

Flues Not Considered Necessary

The basis of each vat is an earthenware pot of about 30 gallons capacity, 22 inches high, 28 inches wide at the top, and tapering toward the bottom. Each pot, which is called "Soochow," is surrounded by brickwork. This brickwork is carried up above the top of the pot wall for a foot or more. The spaces between the tops of these extended pot walls are bricked in, and all brickwork covered with cement. The result is a block of masonry the smooth top of which is broken by the vat openings. Inside this block are spaces left by the tapering bottoms of the Soochow pots. In these spaces some combustible material is kept burning when the vats are in operation to heat the contents to the desired temperature of 20 to 25 deg. Cent. Small holes are left at the floor level to permit introduction of fuel and to provide a draft. Other small openings are provided at intervals in the top of the block to allow smoke to come out. There are no flues to carry the gases out of the building.

For fuel, straw, cotton seeds and similar materials are employed. Usually a brisk fire is not desired or necessary. In Tientsin and other places where there are cold winters, more elaborate heating arrangements are required and a hot coal fire is sometimes necessary.

In most cases of vat construction they are partially sunk in the floor so that the top of the masonry block is 30 inches high.

There are two other kinds of vats in each dyehouse —sour vats for removing the starch sizing and wash vats for the cloth after dyeing. The sour vats are usually of the same construction as the Indigo vats themselves, but with the heating arrangement omitted. They are ranged along one wall of the room.

The size is removed in the sour vats by fermentation. The wet-out piece goods are put into the vat, allowed to remain two days or so, washed and dyed. The sour vats are seldom emptied or cleaned. Just how the bacteria for the fermentation were originally obtained is not clear. If anything goes wrong with such a vat in one dyehouse they borrow a pailful of a good culture from a neighbor.

The wash vats are ordinary Soochow pots without any brickwork around them.

Starting a New Dye Vat

To start a new dye vat, the vat is filled about one-third full of liquor from a vat in active fermentation. The temperature is brought to 20 to 25 deg. Cent. and the Indigo paste fed in in small quantities (one-quarter pound to start). Alkali and bacteria food are added as experience demands. When the first addition of Indigo has been reduced and fermentation is seen to be progressing satisfactorily, further quantities can be added fairly rapidly, care being taken that the vat is not forced. In three or four days, if everything has progressed satisfactorily, the vat is ready for use.

For plain-style dyeing the concentration of Indigo 20 per cent paste to vat liquor is from 1 to 600 to 1 to 300 by weight. For resist styles the concentration may be as high as 1 to 200.

In most dyehouse practice it is usual to keep each vat or group of vats at about the same strength. For a medium-heavy dyeing in a large dyehouse three vats or groups of vats should be used. The cloth from the sour vats would go to the 1 to 600 vat for a few dips, then to one stronger for a few more, and finally end in the strongest.

It is customary to start dyeing very early (4 or 5 o'clock in summer) in the morning. The pieces are dyed to shade, washed and hung on racks in the sun to dry before noon. About 3 o'clock in the afternoon the vats are replenished with Indigo and dyeing assistants, and stirred a few times during the evening. By morning the Indigo is reduced and the vats are ready for use again. This cycle is repeated indefinitely.

The vats are seldom cleaned of sediment so long as

they remain in satisfactory condition. The writer has seen vats which have been in constant operation for two years. The liquor was nearly as thick as gruel.

Manipulation of the Cloth

The manipulation of the cloth during the dyeing is in general the same in all sections of the country. The pieces are never sewn together, but are handled separately. When the size has been removed, the pieces are well washed, squeezed by twisting, and folded. The dyer takes a pile of three or four folded pieces and lays them on the edge of the vat which he proposes to use for the work. He stirs up the vat with a bamboo pole until the sediment is uniformly distributed through it. The end of the top piece of cloth is put into the vat and the rest of the piece is poked under the liquor with a small stick, when all the pieces in the whole are stirred with a pole two or three times during the period of the dip, which usually lasts about ten minutes. The cloth is removed by finding one end and drawing out the piece through a bamboo "pot eye" rested against the side of the vat or through a braided straw ring held in the hand.

In some cases the rope of cloth is further extracted by twisting with two sticks, the material being held over the vat to catch the liquor.

After squeezing, the cloth is opened out by a workman holding each end, and shaken and snapped back and forth to aid oxidation. When oxidized, it is ready for further dips or for washing. Usually the pieces are washed after each dip, but sometimes when heavy navies are to be produced washing takes place only after two or three dips.

When dyed to shade and thoroughly washed, the goods may be topped with methyl violet to give a bronzed appearance, and then starched.

Drying and Finishing

Drying is done on a framework of poles set up in the nearest open space, which may be in a near-by field, over the village street, inside the walls of the dyehouse compound, or even on the roof of the buildings.

When dry, a smooth finish and luster is imparted to the cloth by rolling the folded material between two heavy stones.

Resist Styles

Before bringing these remarks to a close we will deal briefly with the method of dyeing the resist style. The resist is composed of about equal parts of bean flour and slaked lime made into a thick paste. This paste is applied to the dry cloth (previously freed from size) through a stencil. The cloth is laid on a bench in the open width. The stencil is cut from a square piece of tin or heavy oiled cardboard as wide as the cloth. As each piece of cloth is finished it is hung in festoons from small sticks until dry, and is then ready for dyeing.

The cloth is folded in layers about 2 feet long. A small stick, longer than the top of the vat is wide, is attached by means of two hooks to each fold on the left-hand side of the pile of cloth, and the material is lowered into the dye vat.

As previously mentioned, the concentration of Indigo to dye liquor is much higher than for the plain styles. These resist styles are always dyed in deep shades.

Another marked difference between the procedure for the two styles is that with the resist great care is taken that sediment does not come in contact with the cloth. A basketwork with large meshes is lowered into the vat, just previous to entering the material, to form a false bottom. It usually extends halfway to the bottom of the vat.

At the end of each dip the excess dye liquor is squeezed out of the material by very gentle twisting, and each piece of cloth—there are usually three or four pieces of "nesell" per vat—placed edgewise on a rack (which can be pulled over the vat) to drain. During the draining the folds of cloth are shaken out gently by means of the sticks attached to them to give evenness of dyeing and to aid oxidation.

When dyed to shade and dried, the paste resist is scraped off with a blunt knife, the material washed and finished.

Results

Considering the rather crude methods employed, results are surprisingly good. There is little bleeding into the white and only a slight amount of imperfections due to cracking of the paste.

Large quantities of cloth are dyed by this method. In the vicinity of 20 per cent of all Indigo consumed is applied in such a manner. The goods are used for men's and women's outer clothing and for bed covers and hangings.

There are many interesting practices and methods which might be touched upon. There are many precautions which must be observed in this branch of dyeing which we have not mentioned. There are many prejudices and superstitions of which we might write But already these remarks are lengthy and, we fear, rather rambling.

We have attempted, we hope successfully, to sketch a few main points about one of the oldest and most important dyeing industries of a country where secrets of craftsmanship are still handed down from father to son, and where the textile colorist and his laboratory are not yet dreamed of by the great mass of four hundred millions of people.

Para Red Colors on Cotton Goods

Theory of Para Red Dyeing—Preparation of the Goods—Drying—Preparation of the Diazo Bath—The Dyeing Operation—Essential Points

By WILLIAM B. NANSON

CERTAIN organic compounds known as substituted ammonias or amines, when neutralized by acids, are converted into saltlike compounds which react with nitrous acid in a cold acid solution to form diazo compounds or diazo salts. The carrying out of this reaction is known as the diazotizing process.

These diazo salts are soluble in water and when their solution is brought in contact with the solution of the sodium compounds of certain phenols and naphthols, chemical union takes place with the formation of insoluble azo coloring matters which are precipitated in a finely divided condition.

These insoluble azo colors are extensively used for dyeing and printing cotton fabrics. They cannot be applied satisfactorily to animal fibers, as there is one step in the process which requires the use of a strongly alkaline solution.

Theory of Para Red Dyeing

The theory of para red dyeing is something like this: The phenols and naphthols used readily dissolve in concentrated caustic soda solution, forming sodium salts which are soluble in water. When cotton goods are padded in one of these solutions, i. e., with one of sodium beta-naphtholate, it permeates the fiber, and when the material is dried the naphtholate remains upon the surface and within the body of the fiber.

When the material is thus prepared it is passed through a solution of the diazo salt, i. e., paranitro benzene diazo chloride, this solution is rapidly absorbed by the fiber and combines chemically with the sodium beta-naphtholate and the insoluble azo coloring matter "para" or "paranitraniline" red is produced.

The beta-naphthol colors are fast to alkalis and to washing, they do not "bleed"; they are also fast to acids. Their fastness to light is satisfactory for most purposes, being superior, in most cases, to the substantive dyestuffs. The dyeings always rub more or less, especially if the necessary care has not been taken in dyeing. When heated to high temperatures, the color volatilizes slowly. The naphthol itself shows this property, and drying of the cotton which has been prepared with the naphthol solution at too high a temperature, or allowing the goods to be in contact with the surface of the drying cylinders for too long a time, may already cause some of the naphthol to volatilize. Again, we might say here, that too much heat in drying, or, too long a wait between the padding and drying, or, too long a wait after drying, before developing the red color in the diazotized paranitraniline bath will also oxidize the naphthol, turning the prepared material brown, and decomposing and deadening the resultant red color.

The process of dyeing cotton with para red may be considered best under the three following heads:

1. Preparation of the goods.
2. Preparation of the diazo bath.
3. Dyeing of the goods (developing or coupling).

Preparation of the Goods

Previous to the dyeing, the material, if cloth, is prepared with the beta-naphthol by passing it through a padding machine, and then dried at a temperature not exceeding 60 deg. Cent.; this drying is done preferably on a tentering machine, but if drying cans are used the cans must be lapped for some distance with cotton cloth, or, if yarn is being dried, by working in beta-naphthol (using rubber gloves) then wringing, hydro-extracting and drying on a yarn drier. Ordinarily the beta-naphthol padding liquor is prepared somewhat as follows- but we shall show later some modification of this to meet certain requirements:

2¼ pounds beta-naphthol.
2 pounds caustic soda dissolved in a little water.
4½ gallons of hot water; stir until dissolved.

Dissolve separately 5½ pounds of Turkey red oil or 5½ pounds castor oil soap in 2½ gallons of water. Brighter and better shades are obtained by using castor oil soap. Mix the two solutions and dilute with cold water to 12 gallons.

An addition of tartar emetic to the beta-naphthol solution is sometimes made in order to prevent the impregnated material from becoming oxidized and turning a brownish shade during drying. A brighter red is thus obtained. If a precipitate of antimonious oxide (Sb_2O_3) is formed, which is likely to occur, sufficient caustic soda or glycerine must be added to dissolve it and clear the solution.

The best temperature at which to apply the beta-naphthol solution is about 90 to 100 deg. Fahr. and no more padding solution should be made than can be used the same day that it is made, for it does not keep well.

It is of the first importance that all of the machinery and the chemicals used in the padding and drying operations of the beta-naphthol prepare should be scrupulously clean; dirty soap, soda lye or Turkey red oils or water will spoil the brilliance of the red by tingeing the goods

brown. Nothing must be used but chemically pure products and clean *soft* water. Caustic soda lyes containing sulphate of soda, copper or iron salts, oxides or sulphides, not only deaden the red and kill its bloom, but combine with, and precipitate, the various ingredients of the padding solution and thus create a loss of material.

The drying should be carried out at a temperature not exceeding 60 deg. Cent. as soon as possible after the padding operation, and under no condition should the impregnated material be subjected to the direct sunlight, or to acid vapors of any kind or to an atmosphere containing chlorine gas. After the goods are dried it is indispensable to develop the red as quickly as possible, as the prepared cotton rapidly becomes dark colored on exposure to the light, but by adding to the naphthol some mild reducing agent, such as tartar emetic (as previously described) this defect, which is due to oxidation, is greatly diminished.

Preparation of the Diazo Bath

This consists in converting the aromatic amine, or substitute ammonia (paranitraniline), into one of its soluble diazo salts (usually the chloride) and then holding it in solution in a suitable bath until ready to be used in the third step of the process.

The compounds used in its preparation are the aromatic amine sodium nitrite, hydrochloric acid, sodium acetate, ice and water.

The aromatic amine, or substituted ammonia, is usually paranitraniline, or alpha-naphthylamine. These are for the production of reds, and should be used in sufficient quantity to give the desired depth of color.

The sodium nitrite is used in sufficient quantity to liberate enough nitrous acid to carry out the diazotizing process and convert the amine used into a diazo salt.

The hydrochloric acid is used for three purposes: First to neutralize the amine; second, to liberate the nitrous acid from the sodium nitrite; and third, to keep the bath decidedly acid, for, in this condition, the bath is more stable and keeps better. The hydrochloric acid should therefore be used in about three times the quantity necessary to neutralize the amine.

The sodium acetate is added to the diazotizing bath just before it is to be used, in order that it may react with the excess of hydrochloric acid present to form sodium chloride and liberate free acetic acid, hydrochloric acid having been found to be inimical to the best results, if present during the dyeing. The sodium acetate should be added in slight excess of the amount necessary to react with the free hydrochloric acid present. We might say here that the bluish or Turkey red shade of the red is controlled largely by the amount of acetate of soda present; the more of the acetate of soda there is present in the bath and the bluer the shade of the red will be.

The ice. Diazo solutions are extremely unstable, and must, in general, be prepared at a temperature not above 5 deg. Cent. and better at 0 deg. Cent. In order to keep the temperature down, lumps of ice are freely added to the solution, and also to the finished dyeing liquor, otherwise the diazo compound is liable to decompose into a tarry mass that is absolutely useless.

The use of ice is especially necessary in summertime, but in cold weather it may be omitted if the temperature of the diazo bath falls below 50 deg. Fahr. Generally speaking, the colder the bath during the diazotizing process the more stable the diazo compound formed, and this stability is assisted quite measurably if the acetate of soda is not added to the diazotized solution until the latter is required for immediate use.

Not only are diazo solutions susceptible to elevations of temperature, but they may decompose spontaneously on standing a few hours. Indeed the whole process of producing the insoluble azo colors or the fiber must be got through as quickly as possible if good results are to be obtained.

The preparation of the diazo solution requires considerable experience and great care, but a strict adherence to the following directions should give satisfactory results:

Two and one-half pounds paranitraniline.

Seven pounds hydrochloric acid, 36 deg. Tw., are boiled with 1½ gallons water until complete solution takes place, then the solution is poured slowly into 8½ gallons of *cold* water (making 10 gallons altogether) and constantly stirred so that the yellow crystalline precipitate that forms will be in as finely a divided condition as possible. Then add 1 pound 6 ounces sodium nitrite dissolved in one gallon of *cold* water, as quickly as possible, stirring thoroughly. Allow the bath to stand fifteen minutes, when the diazotation will be complete. Just before using, the excess of hydrochloric acid must be neutralized by adding 5½ pounds acetate of soda dissolved in one gallon of water. This combines with the hydrochloric acid to form chloride of sodium and free acetic acid.

Dyeing

This consists in passing the cloth, prepared with the beta-naphthol, in a perfectly dry condition, through the cold diazo solution. The color is produced instantly on passing the padded cotton through this solution and giving it a short exposure to the air, of say a few yards, in order to bring about the full development of the color, the goods being subsequently washed, dried and soaped.

A more bluish shade of red is obtained by replacing part of the beta-naphthol with naphthol sulphonic acid F, up to 8 p.c. The alteration in shade due to this substitution is explained by physical changes, the azo compound of the sulphonic acid being removed by the hot soaping following the developing. Bluer and brighter shades are also produced with castor oil soap than with Turkey red oil.

The presence of alpha-naphthol in the beta-naphthol, which may occur as an impurity must be guarded against as it has a very deleterious effect on the brightness of the red.

(Continued on page 603.)

The Valuation of Dyestuffs by Titration Methods

Disadvantages of Comparative Dyeing Trials—History of Titration Methods—Principles for Successful Work—Basic Dyestuffs—Acid Dyestuffs—Eosine—Direct Cotton Colors—Chrysophenin

By REGINALD B. BROWN and HAROLD JORDAN

A paper presented before the London Section, Society of Dyers and Colourists, and published in the Journal of the Society

THE testing of dyestuffs, compared with the majority of operations by which chemical products are valued, is a less exact and more empirical process. This is true of all branches of the subject, including the recognition of coloring matters, both in commercial form and on the fiber, and their comparison and valuation. The difficulty in exact formulation of methods and tabulation of properties is due to the extremely large number of coal-tar dyestuffs which can be prepared, many differing from one another only to a very slight extent, and to the infinite possibility of combinations, permutations and adulterations to which they may be subjected before making their appearance in commerce.

Disadvantages of Comparative Dyeings

The usual method of testing a dyestuff—putting aside certain crude and rapid operations sometimes adopted in the confectionery and other trades—is to carry out comparative dyeing or staining trials with the colors whose value is to be compared or with a new sample against a standard. This is in some cases the only trustworthy test, but involves the following disadvantages: :

(a) The length of time occupied; an ordinary comparative dyeing trial on wool or cotton cannot well be carried out under two to three hours. This disadvantage when compared with our titration methods disappears in proportion as we are dealing with the simultaneous comparison of several samples, since, once the necessary weighing and preparations are completed, several dyeings can be made at once.

(b) Experimental errors due to unequal heating of the dye baths and rate of evaporation of the dye liquor.

(c) Limitations of the eye in forming judgments.

This last objection is specially prominent in the testing of yellow dyestuffs, where it is frequently impossible to appreciate differences of less than 10 per cent and seldom possible to estimate within 5 per cent. In dyeing comparisons, where it is desired to know the exact relative values, it is often necessary to make several dyeings in order to produce equal depths of color with the various samples.

The methods which we shall describe are, as far as they are at present worked out, chiefly applicable to certain dyestuffs which find an extended application in various branches of industry, and are offered in a large number of brands of varying strength and quality.

Those who were in the dyestuff or dyestuff-using industries before and during the war will readily recall the conditions in the years 1915-1917. The dyestuff situation offered attractive possibilities to merchants, many of whom were unknown to the industry in 1914. The output of British-made colors was still very far from sufficient for our needs and there were always a number of parcels on offer "subject to being unsold." What was a would-be purchaser to do who did not want to buy rubbish but feared to lose a useful color? The early bird secured the prize, and if all the promising samples received were to be tested a rapid method must be found.

If the product did not conform to any known type, as indeed was often the case, a dyeing or its equivalent was indispensable. If the dyestuff corresponded with a known-type a color test in solution or a stain on paper compared with a type might suffice; the former method of comparison may, however, be vitiated by the turbidity of one of the solutions, due possibly to some slight impurity in the intermediates used in manufacture or to the diluting material present. Stains on paper, though fairly trustworthy for most basic dyestuffs, are very much less so for acid and direct colors.

These considerations turned our attention to the volumetric methods which have been proposed for the valuation of coal-tar dyestuffs, and, taking as our basis at first the information on the subject in Knecht, Rawson and Lowenthal's Manual of Dyeing, we soon devised processes for the approximate valuation of a few commonly offered dyestuffs; from these methods were gradually evolved the more exact processes to be described later.

History of Titration Methods

At this stage we may refer briefly to the work which had previously been done in the same field. The history of titration methods for the valuation of coal-tar

dyestuffs begins in 1888, when at a meeting of this society (J. S. D. Col., 1888, p. 82) C. Rawson read a paper entitled "A New Method of Determining the Tinctorial Value of Naphthol Yellow and Certain Other Coal-Tar Colors." In this paper, after pointing out the impossibility of valuing Naphthol Yellow by the ordinary methods to within 5 per cent, the author describes the insoluble precipitate produced by the interaction of Naphthol Yellow S and Night Blue. He dissolves 10 grams Night Blue in 50 c.c. glacial acetic acid and dilutes with water to 1 liter, and also prepares a solution of Naphthol Yellow S containing 1 gram per liter; 10 c.c. of the Night Blue solution are measured into a flask and 30 c.c. of the yellow run in from a burette. The contents of the flask are then poured on to a filter; if the filtrate is yellow, showing excess of the Naphthol Yellow, a further experiment is made with less yellow; if the filtrate is colorless, with more. The trials are repeated until the filtrate seen in a Nessler glass has a faint yellow color. The author considers that with practice three or four titrations should suffice to obtain the correct end-point.

At the same meeting (ibid. 1888, p. 82), Kay and Appleyard describe a similar method in which the reacting dyestuffs are Picric Acid and Night Blue; they also mention the applicability of the method to Crystal Violet.

No further advance is recorded until in 1899 (J. S. D. Col., 1899, p. 148) Baenziger and Vlies published a paper entitled "Notes on the Composition of Some Lakes Formed from Basic and Acid Dyes." They prepared a number of dyestuffs in a pure condition and examined the nature of the precipitates, chiefly with the object of finding whether the end reactions on titration were such as to indicate methods of analysis. They characterize as good in this respect the reactions between Chrysophenine G and Victoria Blue B, and between Methylene Blue and Benzopurpurine 4B; the remainder of those examined were less satisfactory. In the discussion following the paper, Dr. Knecht referred to his attempts to estimate Benzopurpurine 4B by means of Malachite Green. He had found the end-point too indefinite, but had obtained a partial success by spotting on filter paper and looking at the back of the paper.

In the Revue Generale des Matieres Colorantes, 1901, pp. 44-52, is reprinted a portion of the thesis submitted by Alphonse Seyewetz for his doctorate; the section is entitled "Application of the Precipitation of Basic Colors by Acid Colors in the Qualitative and Quantitative Analysis of Coloring Matters." This paper records a vast amount of painstaking work. The principles which Seyewetz claimed to have established and upon which he bases his analytical methods are:

(a) A coloring matter of strongly acid or basic character will precipitate from dilute solutions all coloring matters of the opposite character.

(b) A dyestuff of weaker acid or basic properties will precipitate only a restricted number of colors of the opposite character, the number thus precipitated being greater in proportion as the acid or basic character of the precipitant is more strongly marked.

He bases on selective precipitations of this nature an ingenious scheme of division into groups and gives a series of tables for the recognition of a large number of acid and basic dyestuffs according to these reactions, combined with their dyeing properties and the color of the solutions.

The author also states that certain of these reactions form a basis for quantitative estimations, but refers in detail only to one example; viz., the estimation of Erythrosine by means of a solution of Magenta. He prepared the pure coloring matters and checked the accuracy of his methods by the assay of dyestuffs reduced in strength with known proportions of dextrine and sodium sulphate. He gives, however, no information regarding the recognition of the end-point, and we find it very difficult to understand the feasibility of such a process in a case where the two interacting dyestuffs are so near in color.

In the J. S. D. Col., 1905, p. 9, Knecht refers to a paper in the Bull. de la Soc. Chimique de Paris, by Pelet and Garuti (1904, p. 1094), who estimate Crystal Scarlet or Methylene Blue by their mutual precipitation. They recognize the end-point of the reaction by means of spotting on filter paper and this appears to be the first description of the method exactly as we have carried it out. Knecht expressed a doubt whether the method is sufficiently accurate and Rawson referred the observation of the filtrate in a Nessler glass.

Already in 1903 (J. S. D. Col., 1903, p. 169) Knecht advocated the use of titanous chloride as a volumetric reagent for the estimation of dyestuffs, and described the valuation of azo colors, such as Crystal Scarlet and Orange II, either by direct titration or by adding excess of titanous chloride and titrating back with iron alum until a drop of the liquid gives a red spot with potassium sulphocyanide.

In subsequent papers (J. S. D. Col., 1905, p. 11, and 1905, p. 285) Knecht and Hibbert describe continued researches on the titanium method; by the addition of Rochelle salt to the dyestuff solution they are able to obtain a definite end-point by direct titration in most cases.

It is possible that the titanous chloride method is in practical use by color makers or users, but we ourselves have not made use of it, chiefly because for good results it is necessary not only to preserve the titanium solution out of contact with air but also to titrate in an atmosphere of carbon dioxide or other inert gas, and we wished to avoid even this slight complication of the process.

We decided to concentrate on methods based on the mutual precipitation of acid and basic colors, and at an early stage we established the following principles for successful work:

1. The dyestuff to be estimated and the volumetric reagent selected must in solution be of quite different colors; e. g., blue with red, green or blue with yellow.

2. The acid color should be run into the solution of the basic dyestuff; the reverse method seldom gives a useful result.

3. In determining the end-point a quick judgment must be arrived at.

We work with dyestuff solutions containing 1 gram in 500 c.c. and recognize the end-point by the appearance in a spot made on filter paper of a ring of the color of the precipitating solution. We can by this method estimate tinctorial value to within 2½ per cent, which is much nearer than a practiced eye can judge in the case of comparative dyeings.

Referring now in detail to a few cases where we have established useful and accurate methods of valuation:

Basic Dyestuffs

Auramine.—This was one of the first dyestuffs for which we employed a volumetric method. After trials with various acid dyestuffs as precipitating agent we finally selected Indigo Carmine as the most suitable; our present method is to prepare a solution of the Auramine slightly acidified with acetic acid and containing 1 gram dyestuff in 500 c.c. We titrate with a solution of a concentrated Indigo Carmine also 1 in 500. Spots on filter paper at intervals show a yellow ring which gradually disappears as the Indigo Carmine is added. The end-point is clear and is marked by the first appearance of a blue ring.

To test the accuracy of the method, three samples were prepared for us of a standard Auramine reduced with definite proportions of dextrine, common salt and sodium sulphate, respectively, with the following result:

VALUATION OF AURAMINE

	Found (per cent)	Theory (per cent)
Auramine O	100.00	100.00
Sample A (Dextrine)	81.40	80.00
Sample B (Salt)	64.60	66.66
Sample C (Na_2SO_4)	72.85	72.72

Various commercial products offered as Auramine O gave relative values of 112, 100, 128, 92. Stains on paper and dyeings on tannin mordant in which the quantity of dyestuff taken is inversely proportional to the strength as found by the titration method show an equal depth of color.

Brilliant Green and Malachite Green.—The strength of solutions is the same as with Auramine. Indigo Carmine is excluded as a precipitating agent, as the color is too near that of the green. We found Orange II the most suitable acid color for titration, but the end-point is not definite enough. Eventually we modified the process by employing the acid dyestuff in conjunction with a tannin solution, and this method, which we do not think has been employed before, has been found of the greatest service in assaying many basic colors. In this instance we prepare a solution containing 1 gram Orange II, 2 grams tannic acid and 2.5 grams sodium acetate per liter. We titrate 25 c.c. of the green solution (1 in 500) with this mixture until no further precipitate is formed, this point being indicated by the appearance of a distinct orange ring. The precipitate formed in the presence of tannin is more definitely granular in form than when Orange alone is used, and allows the orange ring to be seen more clearly. The final point is quite definitely brought about by an addition of 0.5 c.c., which corresponds to a difference of 2½ per cent in the concentration of the dyestuff.

Magenta.—The method employed is the same as for Brilliant Green, but the acid dyestuff used in conjunction with tannic acid and sodium acetate is Indigo Carmine. The end-point is shown by a faint greenish-blue ring.

Methyl Violet.—Methyl Violet can be estimated without the necessity of adding tannin. The precipitating agent is Naphthol Yellow S; we employ a concentrated commercial brand of this dyestuff, such as the brand known as "SXX," and obtain a clear end-point shown by a black precipitate with a yellow ring.

Methylene Blue.—Up to the present we have titrated with a solution of a concentrated Tartrazine containing also tannic acid and sodium acetate. Some of our recent work, however, indicates that a better result may be obtained by adopting the process of Pelet and Garuti already referred to and substituting Crystal Scarlet for the Tartrazine. We do not, however, obtain a good end-point without the addition of tannic acid to the Crystal Scarlet solution. When the dyestuff is used alone, the edge of the spot is blurred and a blue zone persists even after the first appearance of the red ring. With the addition of tannic acid and sodium acetate, the edge of the spot is ragged but the red appears distinctly without a blue zone. In connection with this dyestuff we attempted an estimation with tannic acid alone. As the tannic acid solution is almost colorless, we could only determine when this reagent is in excess by spotting on to filter paper which had been previously impregnated with an iron salt. When tannic acid is in excess a dull grayish violet rim is seen. The neutral tint of this rim renders it somewhat difficult to observe and not very suitable as the indication of an end-point.

It was interesting to observe that the addition of tannin to the acid color solution causes only a slight decrease in the quantity of precipitating agent required, showing that in the mixed solution the acid dyestuff precipitates the greater amount of the basic color. The figures are:

Crystal Scarlet alone.....19 c.c. required
Tannic acid alone.....37 c.c. required
Crystal Scarlet and tannic acid.....17 c.c. required

Rhodamine (B and 6G).—We have tried a large number of acid dyestuffs as precipitating agent, but have failed so far to find the perfect reagent. When Indigo Carmine, which is an excellent precipitating agent for other red basic dyestuffs, is used even in the presence of tannin, no definite end-point can be recognized—the pink rim shown at first gradually changes through violet to a greenish-blue. The best result we have found so far, and one which has proved capable of practical use, is titration with Orange MNO (concentrated Metanil Yellow) and tannic acid by the method already described.

Saframine.—This can be assayed with Indigo Carmine, tannic acid and sodium acetate.

Victoria Blue B.—This dyestuff is titrated with concentrated Tartrazine alone. The color is gradually precipitated and finally leaves a clear liquor in which a slight excess of the yellow precipitant is easily recognized.

The valuation of acid colors is, of course, the same process as the estimation of basic colors, the quantity of acid dye in solution required to precipitate a given weight of the basic dye in solution being an index of the color strength. We have only established on a definite practical footing the valuation of four acid dyestuffs—viz., Orange II, Orange I, Tartrazine, and Naphthol Yellow S—and in all cases we employ Victoria Blue B or Night Blue as the basic color.

Orange II.—The valuation of Orange II may be taken as typical. A solution of the sample to be valued (1 gram in 500 c.c.) is run into 25 c.c. of a Victoria Blue solution of the same strength. The blue solution gradually loses its brightness and a blue-black precipitate is formed. The first appearance of an orange rim indicates the end of the reaction and the strength of the orange sample is, of course, inversely proportional to the number of cubic centimeters used.

We have taken this case of Orange II for illustrating the use of our method in a rapid commercial valuation. A few days ago we desired to make a purchase of Orange II and received offers and samples from six different sources. These were all compared with a standard sample of Orange II of Badische make, with the following results:

VALUATION OF SAMPLES OF ORANGE II

	Proportionate Quantity of Orange Solution Required	Relative Concentration Standard = 100 10,000
	x	x
Sample A (standard).....	100.0	100.0
Sample B	87.0	114.9
Sample C	104.3	95.9
Sample D	95.6	104.6
Sample E	104.3	95.9
Sample F	117.4	85.2
Sample G	127.0	77.8

In order to check the value of these results as indicating the real tinctorial power of the samples, we have prepared two series of comparative stains on filter paper. In the first series, 0.1 gram of each sample was dissolved in 100 c.c. of water and stains made with each by pouring the solution on to the paper and drying all the papers at the same temperature. In the second series the weights of the samples dissolved in 100 c.c. of water were inversely proportional to the color strength as found by the titration test; these stains should, of course, all appear of equal intensity, though they might show slight differences in tone or purity of shade. It will be seen that this is indeed the case and it is difficult to recognize any difference in depth of color between the stains in this series.

Two other advantages of our methods may be referred to at this point. The figures in the first column of the above table allow of a ready calculation of the relative price value. These figures, or even the actual number of cubic centimeters taken, merely require to be multiplied by the price in pence to show the relative values—the resulting figure is, of course, inversely proportional to the actual color cost, the lowest number representing the highest value. Thus:

	I Titration Fig.	II Price per Lb.	Product of I and II
Sample B	87.0	2/—	2,088
Sample C	104.3	1/11	2,399
Sample D	95.6	1/7	1,816
Sample E	104.3	1/6	1,877
Sample F	117.4	1/6	2,113
Sample G	127.0	1/9½	2,730

Sample D, other properties being equal, thus shows the best value.

The method permits further of a permanent record of the color value of a sample without the necessity of preserving a bulky dyeing or a stain liable to alteration by the passage of time. Should we, for example, again have occasion to purchase Orange and be furnished with samples of new products, we need only test these against the original standard and compare the results with the records of our former tests; to insure exact comparisons it is essential that the standard color should always be titrated at the same time as the new samples.

Eosine

For the reason already given—viz., the similarity of the color solutions—we dismissed the method of Seyewetz, titrating with Magenta. Our best result in the valuation of Eosine dyestuffs is obtained by running the Eosine solution into Malachite Green, and we are able in this way to distinguish differences of 2 per cent between Eosines of similar shade.

We have investigated briefly the possibilities of the suggestions of Baenziger and Vlies for the estimation of Benzopurpurin with Methylene Blue and Chrysophenin with Victoria Blue B, but can hardly claim to have worked out completely the best method for the valuation of these dyestuffs.

Benzopurpurin 4B.—We first attempted the direct titration of Methylene Blue (without addition of acetic acid) with Benzopurpurin 4B. In this titration the blue rim shown by the earlier spots appears more and more slowly until, as the end-point is passed, a ragged violet rim takes its place—even a considerable excess of Benzo does not show a definite red ring.

We next tried the reverse operation and titrated Benzopurpurin 4B with Methylene Blue. Here also no definite end-point can be distinguished, as there is a long interval between the disappearance of the red and the appearance of the blue ring.

With Victoria Blue also a direct titration is not feasible, but we achieved a certain amount of success by adding an excess of Victoria Blue and titrating back with Orange II. We found that an addition of alkali to the Benzopurpurine sclution is essential, and that this solution should be near boiling temperature when added to the Victoria Blue.

A solution of the Benzopurpurin sample containing 4 grams per liter—i. e., double the usual strength—is prepared. To 20 c.c. of this solution we add 20 c.c. of a soda ash solution (5 grams per liter) and heat this nearly to the boil before adding it to 50 c.c. Victoria Blue solution of the usual strength. We then titrate with Orange II (2 grams per liter) until the spot shows an orange rim.

Four samples of reduced Benzopurpurin were prepared for us, consisting of:

A.....2.00 grams Benzopurpurin 4B
1.00 gram soda
B.....2.00 grams Benzopurpurin 4B
1.00 gram common salt
C.....2.00 grams Benzopurpurin 4B
0.50 gram salt
0.50 gram soda
D.....2.00 grams Benzopurpurin 4B
0.75 gram salt
0.50 gram soda

A blank test was first made without the addition of Benzopurpurin 4B, and we found that 50 c.c. Victoria Blue required 22 c.c. Orange II. The four samples were then tested against standard and gave the following result:

VALUATION OF BENZOPURPURIN 4B

Sample	C.C. Orange II	*Difference	Found (per cent)	Theory (per cent)
Standard	7.00	15.00	100.00	100.00
Sample A	11.50	10.50	70.00	66.66
Sample B	12.00	10.00	66.66	66.66
Sample C	11.50	10.50	70.00	66.66
Sample D	11.25	10.75	71.66	72.72

*Orange II corresponding to Victoria Blue pptd. by Benzopurpurin 4B.

The method is thus approximate and the result evidently varies with the alkalinity of the solution—we propose to investigate this reaction further. A trial made with Auramine in place of Victoria Blue and titrating back with Indigo Carmine indicates greater accuracy and promises to furnish a useful practical method.

Chrysophenin.—We were not able to achieve success by direct titration with Victoria Blue, but as in the case of Benzopurpurin fair results were obtained by adding excess of Victoria Blue and titrating back with Orange II. An addition of soda improved the accuracy and by titrating hot instead of cold a still greater improvement was shown. Here also it now appears that Auramine may with advantage be substituted for the Victoria Blue. In this case we titrate back with Indigo Carmine; the end-point is clear and it is not necessary to work with hot solutions.

Two samples were prepared with:

A.....1.7 grams Chrysophenin (Cotton Yellow CH)
0.3 gram salt
B.....1.5 grams Chrysophenin
0.5 gram soda

and were tested by the above methods with Victoria Blue and also with Auramine. The results shown were:

VALUATION OF CHRYSOPHENIN

	With Victoria Blue	With Auramine	Theory
Standard	100.0	100.0	100.0
Sample A	84.8	87.2	85.0
Sample B	70.0	74.4	75.0

We are aware that the various methods described in this paper might with very great advantage be worked out in greater detail. To take one point among many, we find that concordant results are only obtained by working within certain limits of concentration.

If it is desired to value products which are very much stronger or weaker than the usual standard taken for comparison, it is well to prepare a solution approximating to the usual strength—e. g., in testing a sample of Rhodamine B extra, which is usually five times as strong as the ordinary Rhodamine, the solution of this dyestuff should contain 0.4 gram per liter instead of 2 grams per liter.

Another question which naturally arises is the effect of slight differences in the shade and therefore in the constitution of the products tested on the result of titration. Such effects will, of course, depend on the molecular weights of the coloring matters in question, and can only be investigated with any degree of precision with specially purified dyestuffs.

We are still in the dark regarding all the possibilities and the limitations of these titration methods, and the theoretical basis of many of the observed results

is still puzzling us. Much of the work we have described must appear unfinished, and most of our methods are temporary makeshifts pending the fuller knowledge which patient work will bring in time, but we considered that it might add to the interest of our paper and perhaps stimulate others to work in the same field if we did not confine our descriptions to methods accepted as fully satisfactory.

The subject we have dealt with was stigmatized in a discussion at a meeting of this society some years ago as an unprofitable one, but our personal experience has proved it of considerable value at any rate in our own work. Several of the methods described have been in regular use in our laboratory for four or five years and the results have never yet led us astray. Our aim has been, as already stated, to work out rapid methods of estimation which can be adapted at once for technical and commercial purposes with the hope that a more ample leisure later on may allow of a more scientific investigation of the laws which govern the facts we have observed.

Discussion

S. Verrinder asked whether in titration of Methyl Violets with Naphthol Yellow S variations in shade did not give rise to error.

The lecturer said that titration gives accurate determinations when the shades of violet are not very dissimilar.

J. T. Jackson asked whether the addition of sodium acetate alone to the precipitating agent had been tried.

The lecturer replied in the negative. (Note—This has been tried since and found to be not effective without tannic acid.)

F. W. Walker considered that titration methods were satisfactory for the relative valuation of samples of dyes like Orange II. Did the lecturers consider that titration was effective in the case of dyes that depend for their value on brightness; for example, Rhodamine? Also, how were bronzing difficulties overcome; for example, in the case of Victoria Blue? Could partially insoluble dyes be titrated at high temperatures?

The lecturer replied that titration was not claimed to be effective in comparing brightness, but the method was always effective for preliminary strength determinations. Bronzing is avoided by the addition of a little acetic acid. For partially insoluble dyestuffs, sodium carbonate is found to be beneficial, also titration can be carried out at high temperatures; but comparative tests must always be carried out at the same temperature.

Mr. Verrinder asked how the impurities in dyestuffs affected the end-point, and the lecturer replied that the effect was very slight and not nearly so great as in actual dyeing trials.

Mr. Walker asked whether the end-point was more visible through the reverse side of the filter paper, and the reply was that the end-point can be seen quite clearly on the face of the paper, especially when damp.

In reply to further questions the lecturer stated that the work had not yet been successfully extended to blacks or neutral dyeing acid colors. Various qualities of filter papers of different degrees of hardness acted somewhat differently; for example, hard paper gave spots with less ragged outlines. The Lovibond tintometer was only useful for detecting the presence of other dyes and not for valuation of strength.

FRENCH TO SELL PART OF SEIZED RUHR COLORS

The U. S. Department of Commerce has received from Commercial Attaches Jones and Herring, of Paris and Berlin respectively, a report to the effect that dyestuffs seized in the Ruhr district, 6,000 tons net, will be drawn upon by the French to the extent of some 2,000 tons for their own needs, and the balance will be sold for export. The report states:

"The Union des Producteurs et des Consommateurs des Matieres Colorantes probably will conduct the sales, though it is still undecided whether all sales shall be through one agency or sales by the French and Belgians separately. The present feeling is that there is no danger the sales will upset the market, as the union has previous experience in handling reparations dyes and demand from many countries makes good prices probable.

"Representatives of American consumers have orders for 500,000 pounds."

Transportation throughout the occupied area is demoralized, and unemployment is a big problem, as the Government support cannot be maintained indefinitely.

"On the whole," the report declares, "chemical and dye stocks are not likely to be as important a factor in international trade when the deadlock ends as might be supposed. Plants outside British territory are disinclined to produce for stock which later may be commandeered, and the abnormal demand from the consuming industries of unoccupied Germany will tend to offset abnormal stocks in the British zone. Furthermore, stocks, belonging to plants in the British zone were previously established in unoccupied Germany, and are now being drawn upon so that net stocks at the end of the crisis will be proportionately reduced.

"The chemicals and dyestuffs seized have been taken under the alleged authority of the reparations agreement. American buyers report that the French announced as their intention the seizing of the amounts of chemicals and dyestuffs due from the Badische Anilin and the Hochst plants at Ludwigshafen and Oppau, plus such amounts as should have been delivered by Berlin and Frankfort plans, plus enough to cover the cost of the seizures."

Education and Research in the Textile Industry[1]

Education in General—Objects—Methods—Results—Technical Education, with Special Reference to Textile Education—Textile Research

By A. J. TURNER, M.A., B.Sc.

(References given in this article will be found at the end.)

IN dealing with the subject forming the title to this paper I propose to consider it in three parts, viz.: (1) Education in general; (2) technical education in particular, with special reference to textile education; (3) textile research. It is the interrelation of the second and third which is of primary interest.

1. Education in General

We may distinguish between (a) objects, (b) methods, (c) results.

(A) Objects

To introduce this subject I cannot do better than quote what John Ruskin said in Manchester in 1864, when delivering the first of his lectures, afterwards known as "Sesame and Lilies": "It happens . . . that I receive many letters from parents respecting the education of their children. In the mass of these letters I am always struck by the precedence which the idea of a position in life takes above all other thoughts in the parents' minds. The education befitting such and such a 'station in life'; this is the phrase, this the object, always. They never seek an education good in itself, but an education which shall keep a good coat on my son's back; an education which shall enable him to ring with confidence the visitors' bell at double-belled doors; education which shall result ultimately in establishment of a double-belled door to his own house—in a word, which shall lead to advancement in life." Such was what appeared to Ruskin to be the parents' view of the object of education in general. Although this utilitarian view is, no doubt, widely held by parents even to-day, it has not been shared by most educationists. At the same time educationists have by no means always agreed among themselves as to what is the ultimate object of education. Indeed, the statements of the objects of education have been as numerous as the methods which have been advocated to attain those objects. The following are a few of the definitions:

1. "To draw up from the region of shadows into the daylight of realities men of superior natural aptitude"; the aim to which education should be directed being "to train the intellect to the contemplation of realities, and not the senses to the observation of things."—Plato.

2. The preparation for complete living.—Herbert Spencer.

3. Preparation of a perfect citizen [cf. Herbert Spencer: "Education, of whatever kind, has for its proximate end to prepare a child for the business of life, who, while he is well conducted, is also able to acquire funds requisite for the bringing up of a family"].

4. Adaptation to environment.

5. Perfection of our nature.—Rousseau.

6. Sound mind in a sound body.

7. The harmonious development of all the faculties—observation, memory, reasoning, etc.

8. The development of the power to discriminate between good and evil (morality), truth and error (science), beauty and ugliness (art).

9. The strengthening of character and the development of intelligence in preparation for life.—Education Act, 1870.

10. The transmission of life from the living through the living to the living.

11. To repair the ruins of our first parents by regaining to know God aright.

12. "Education is not the storing of the compartments of the mind, but the development and training of faculties already existing. It proceeds, not by the presentation of lifeless facts, but by teaching the student to follow the different lines on which life may be explored, and proficiency in living may be obtained; it is, in a word, guidance in the acquiring of experience."[2]

There is no doubt a great deal to be said in favor of each of these definitions of education. Each of them, at any rate, presents a number of points of interest. However, I intend to follow Adams,[3] who reduces most of them to a common denominator, as follows: "The aim of the whole process of education is: (1) Self-realization by the educated (person being educated)—this is based on the nature of the educand; (2) development of a many-sided interest in the educand—this has a definite relation to the reaction between the educand and knowledge."

Instead of the expression "many-sided interest," Dr.

[1] Lecture given before the Textile Teachers' Association.

Maxwell Garnett[4] prefers "single-wide interest," in order to emphasize the essential unity of the interest. This is reminiscent of the views of believers in "integral education," who insisted on the unity of mental life—in intelligence, as well as in knowledge.

(B) Methods

It is pointed out by Adams that the process of formal education includes the following factors: (1) Education is a bipolar process in which one personality acts upon another in order to modify the development of that other. (2) The process is not only a conscious but a deliberate one. The educator has the clearly realized intention of modifying the development of the educand. (3) The means by which the development of the educand is to be modified are twofold: (a) Direct application of the educator's personality to the personality of the educand; (b) the use of knowledge in its various forms. The communication of knowledge tends to play the predominant part.

But if we leave these general ideas to consider particular methods, we are again confronted by a diversity of views. The methods advocated tend to vary according to the philosophy and psychology believed in —the former determining the view of the object of education and the latter being concerned with the mental make-up of the individual in whom this object is to be achieved. I do not propose to discuss educational methods in detail. I may, however, be allowed to say just a few words on two methods whose fundamental principles apper to be diametrically opposed. On the one hand, there have been those who have pinned their faith to the value of discipline in education; they would have the subject matter be as dry-as-dust as possible—the more barren of interest to the educand the better, the theory being that his mind, by forcible feeding on uncongenial food, receives a discipline which is invaluable, and which does, in fact, constitute his education; the knowledge which is gained is, in itself, of no importance. On the other hand, there are those who believe in the doctrine of interest—that the best method of education depends on arousing the interest of the educand in the subject matter of his education, and that the knowledge gained is, in itself, of the highest importance, quite apart from the discipline involved in acquiring it. This is a statement of the case from the two extreme points of view. For my own part, I believe that while a great deal depends upon the personalities and capacities of the individual educator and educand, respectively, most subjects can be taught so as to discipline the mind; and that if the subject matter is interesting, and the knowledge gained is, in itself, useful to the educand, the educator is possessed of a very valuable asset in his work.

It will be observed that what has been said relates more particularly to the development of a single-wide interest. But the educational method must do more than this; it must also lead the educand to self-realization, for which real effort on his own part is essential. As to how this is to be secured, I cannot do better than quote from Locke on "Education": "Converse with books is not the principal part of study; there are two others which ought to be joined with it, each whereof contributes its own share to our improvement in knowledge, and those are meditation and discourse. 'Reading,' methinks, is but collecting the rough materials, amongst which a great deal must be laid aside as useless. 'Meditation' is, as it were, choosing and fitting the materials, framing the timbers, squaring and laying the stones, and raising the buildings; and 'discourse' with a friend (for wrangling in a dispute is of little use) is, as it were, surveying the structure, walking in the rooms, and observing the symmetry and agreement of the parts, taking notice of the solidity or defects of the works, and the best way to find out and correct what is amiss; besides that, it often helps to discover truths and fix them in our minds as much as either of the other two."

(C) Results

When we come to consider the results of education, various questions present themselves. How are we to determine the results of any method adopted? How are we to tell whether our object has been achieved? Obviously we need some form of measurement, and, truth to tell, there is no form of measurement available. Great strides have been made in recent years in vocational selection, but how far education is responsible for the qualities measured is, of course, another matter. The fact is, we can only express the results of the education in general terms. Certainly education should produce, so far as the educand's natural limitations will allow, those general results already alluded to among the objects of education. To state some of them: Useful knowledge should be imparted, but not only knowledge—the educand should gain in wisdom and understanding; this is the development of intelligence. We should also get strengthening of character—by the disciplinary effort of the subjects studied leading to these results. Power of concentration should have been gained, and that thrill of pleasure experienced which comes from the conquest of a difficult problem, leaving the student sighing for fresh worlds to conquer. And, what is very important, the student should become more adaptable to new circumstances than ever he would have been without his education. This matter of adaptability one would like to dwell upon, but I must pass on to other parts of my paper.

2. Technical Education, with Special Reference to Textile Education

Now we come to speak more particularly of technical and textile education. Although such education

has a long history, it only became widespread in this country as a result of the Technical Instruction Act of 1889. This defined technical instruction as "instruction in the principles of science and art applicable to industries, and in the application of special branches of science and art to specific industries and employments." Note the word "instruction." We now aim at "education," which is a bigger thing than instruction; we want to induce the student to attain self-realization and a single-wide interest, chiefly through a textile education. Now, from its nature textile technology is largely descriptive. Yet this makes it all the more necessary for losing no opportunity which the subject allows for attaining the two ends mentioned. Can we say that it does at present function in this way? I do not suppose that anyone will maintain that it does so to perfection. Perhaps I may be allowed to quote what the aforementioned Committee on the Teaching of English think about the matter:[5] "We believe in technical education, and we yield to none in our admiration for what it has done and is doing for this country; but we hold that, nevertheless, it is at present imperfect and lopsided—is not, indeed, rightly 'education' at all." The remedy they propose is the introduction of English "not as an excrescence but as an integral part of the students' courses." This "would not only react immediately upon their technical studies to their immense advantage, but would give to technology the soul which it now lacks, and in the end perhaps bring a new spirit into business at large." Personally, I agree with this dictum to the extent that for a complete education there must be a humanistic element present.[6]

Leaving on one side for the moment the larger question whether it is possible so to shape a textile course that it includes the humanistic element necessary for complete self-realization and for the single-wide interest, one has to face the question whether textile education is or can be made a scientific education. I have heard criticism from various quarters that it is not. At any rate, my personal belief is that there is no reason at all why it should not be so, and I feel confident that in this you will all agree. We still, however, have to face the fact that textile technology is largely descriptive. Some critics say that textile education confines itself to imparting isolated mill facts and empirical rules relating thereto. There is no doubt that any such teaching is not education. Solely to concern oneself with "pumping facts" into the student produces the same kind of results as overloading a carding engine with cotton—the material is damaged, the product is unsatisfactory, and the machine is discredited. Thus, to teach isolated mill facts is a waste of time. The place to teach only mill facts is the mill itself. The whole atmosphere there is favorable to their being acquired in the proper way and in their proper setting. The student's mind is not needlessly encumbered with a lot of other mill facts which have no perceptible relation to his own work.

On the other hand, let me not be misunderstood. The teaching of isolated mill facts is bad, not because they are mill facts but because they are isolated. Education begins when, and only when, all these isolated facts are shown in their proper relation to one another and to the whole structure of textile technology. In the textile industries themselves attention to detail is all-important. One must never underrate the very practical importance of detail. It is by attention to detail that the textile industries live; it makes all the difference between prosperity and bankruptcy. And likewise, on the educational side, the more a subject is studied the more does detail come into the field of vision; and the more detailed the study the more interesting and the more living does it become. But what is essential is that details, by the educational process, should be co-ordinated. "The knowledge that counts, the knowledge that is power, is not mere acquaintance with facts, but experience of facts in their relation to each other."[7] The student should have in his mind a sort of genealogical tree wherein all his details are linked up together; in short, he must acquire a unity of knowledge in textile technology. And from the standpoint of a complete education this unity is only one of a number of unities all linked together to form the single-wide interest.

To obtain this unity of knowledge in textile technology a student must understand something of fundamental sciences. By starting with and developing from these, a textile education may be made a scientific education. To arouse and maintain the interest of students in these fundamental sciences, the teachers will naturally make frequent references to their application in the textile industries. Nevertheless, it is unreasonable to expect a teacher to restrict himself to examples of this kind. Such a limitation is not only unnatural—it defeats the ultimate purpose of education of building up a single-wide interest. Moreover, while it is important that the teacher of a fundamental science should not divorce the science from textile technology, it is equally important that the teacher of textile technology should not do so. He, for his part, should point to the operation of scientific laws and principles wherever possible in his description or discussion of textile mechanism or processes.

So much for the educational process in textile technology as leading to the development of a single-wide interest. But what of the development of self-realization? It is proverbially more blessed to give than to receive, but the teacher must restrain his natural tendency only to give; he must, in fact, demand something from the student. He must encourage the student to allot some time to "meditation and discourse." This entails that the student's time-table shall not be so full that he has "no time to think"; and that some provision is made, either within a class or outside it, for discussion. Perhaps one of the best incentives to self-realization is the writing of essays. To compose a good essay, with only the title given, necessitates

much hard thinking on the part of the student: he has to exercise his mind on the selection or rejection of material; he has to plan the essay as a whole and in its parts, maintaining a logical sequence and yet preserving the coherence of the whole; and, above all, he has to display the results of his reading, meditation and discourse. It is not easy to mention any better aid to the development of self-realization.

3. Textile Research

The next question we have to face takes us to the domain of research. Does the present-day textile education equip a student for bringing about progress in the textile industries? Does it, in other words, fit him for carrying out research? Let us turn to the present state of the industry. It is commonly asserted that, with only one or two exceptions, there have been no "great" inventions for a century. Some people have deduced from this that education, invention and research must in future proceed along new lines; that the old lines are worn out. In seeking to controvert this view it is not sufficient to point, as has been done, to the lengthy lists of patents frequently published, because the question then becomes more insistent: What becomes of these patents? There is no doubt that at the present time we are breathing a new atmosphere of research. One cannot help wondering if this is a similar sort of atmosphere to that which prevailed in the magic years round about 1770. Are we again on the threshold of new developments on new lines? It is possible that we have now reached a stage where intensive research will lead to great progress, the stage indicated by Herbert Spencer when he says: "In each branch of instruction we should proceed from the empirical to the rational. During human progress every science is evolved out of its corresponding art. It results from the necessity we are under, both individually and as a race, of reaching the abstract by way of the concrete—that there must be practice and an accruing experience, with its empirical generalization, before there can be science. Science is organized knowledge; and before knowledge can be organized, some of it must be possessed. Every study, therefore, should have a purely experimental introduction; and only after an ample fund of observations has been accumulated should reasoning begin." With this quotation I may couple one from Dr. Lawrence Balls:[9] "The case for research in the cotton industry is thus initially a plea for reducing the incoherent mass of details to a small number of generalizations which the human mind can easily handle, so that apprentices may be able to acquire as much technical knowledge in a few months as they now waste years upon, so that discussions of technical problems can be carried on in the shorthand of general science (we have heard an explanation last twenty minutes which a little elementary physics on both sides would have condensed to a single sentence), and so that there may be material available for constructive work, and, ultimately, for the initiation of real industrial research with its limitless possibilities."

The various textile research associations are now busily at work; research is also being vigorously prosecuted elsewhere, and doubtless in the next few years we shall know the answer to our question as to whether a new era of progress is being inaugurated. But let us return to the previous question as to the adequacy of the present textile education as a training for research. According to some, it is desirable, in view of the fact that textile research depends so much on fundamental sciences, that training in textile technology should be post-graduate, following a degree course in science. I must confess to much sympathy with this view, but we encounter the almost insuperable difficulty that the ordinary student cannot afford to spend so much time as this would entail in acquiring his training. Properly trained students, however, should be able to understand the research papers being published at the present time, and be able also to undertake research themselves. But not if the only result of their training has been to fortify them with isolated mill facts and rules. Perhaps I may be forgiven for quoting Herbert Spencer once more:[10] "Between a mind of rules and a mind of principles there exists a difference such as that between a confused heap of materials and the same materials organized into a complete whole with all its parts bound together, of which types this last has not only the advantage that its constituent parts are better retained, but the much greater advantage that it forms an efficient agent for inquiry, for independent thought, for discovery—ends for which the first is useless."

Note that point: the properly trained student is thus naturally equipped for research. At least one function of education is to cause the mind to develop on rational lines—to develop "the thinking habit"; and the thinking habit is needed for research. By indicating how processes of reasoning have solved problems in the past, and by developing the reasoning faculty, we teach how to face up to new problems as they arise, and at the same time provide the instrument for solving the problems. Thus two ends of education are made possible—that of fitting a man for his environment and that of so equipping a man that he may modify his environment. Knowledge is shown to be a living tree whose branches are ever extending their ramifications. Follow any branch to its end and you come to ask questions to which the answers lie in the unknown region beyond. This is how and where research begins. And the methods to be used at the methods of which a sound education should leave the student master. To conclude in the words of Huxley:[11] "The method of scientific investigation is nothing but the expression of the necessary mode of working of the human mind. It is simply the

mode at which all phenomena are reasoned about, rendered precise and exact. There is no more difference, but there is just the same kind of difference, between the mental operations of a man of science and those of an ordinary person, as there is between the operations and methods of a baker or of a butcher weighing out his goods in common scales, and the operations of a chemist in performing a difficult and complete analysis by means of his balance and finely graduated weights. It is not that the action of the scales in the one case and the balance in the other differ in the principles of their construction or manner of working, but the beam is set on an infinitely finer axis than the other, and, of course, turns by the addition of a much smaller weight.

"You have all heard it repeated, I dare say, that men of science work by means of induction and deduction, and that by the help of these operations they, in a sort of sense, wring from nature certain other things which are called natural laws and causes, and that out of these by some cunning skill of their own they build up hypotheses and theories. And it is imagined by many that the operations of the common mind can be by no means compared with these processes, and that they have to be acquired by a sort of special apprenticeship to the craft. To hear all these large words you would think that the mind of a man of science must be constituted differently from that of his fellow men; but if you will not be frightened by terms you will discover that you are quite wrong, and that all these terrible apparatus are being used by yourselves every day and every hour of your lives."—The Textile Manufacturer.

References

[2] Report of Committee on the Teaching of English in England (1921).
[3] "Evolution of Educational Theory," p. 39.
[4] "Education and World Citizenship," p. 22.
[5] "Education and World Citizenship," p. 153.
[6] Cf. Address by Sir Thomas Holland, "Humanism in Technical Education"; Nature, March 17, 1923, p. 376.
[7] Adams: "Evolution of Educational Theory," p. 188.
[8] "Education," p. 69.
[9] "The Nature, Scope and Difficulties of Industrial Research: Tenth International Congress of International Federation of Master Cotton Spinners," Zurich, 1920, p. 436.
[10] "Education," p. 55.
[11] "Man's Place in Nature."

The Titration of Sodium Formaldehydesulfoxalate[1]

Inaccuracy of Jellinek's Method—Knowledge of Nature of Impurities the Goal—Titration of Sample in Three Different Ways—Calculation—Analysis of Sulfoxalates

By BERNARD SALKIN

As late as February, 1922,[2] it has been claimed that it is practically impossible to titrate sodium formaldehydesulfoxalate by two different methods and obtain equivalent results. Furthermore, two very simple methods—that of titration with an alkaline copper sulfate solution, and an idometric titration—are displaced in favor of more complicated methods. This can be explained by the fact that the procedure outlined by Jellinek[3] is not conducive to correct and uniform results. As the result of experimentation a method has been worked out which will not only give uniform results in the determination of the main product, but will also indicate the kind and the amount of the impurities present.

Just as with sodium hyposulfite, the titration of sodium formaldehydesulfoxalate can very easily—and best—be carried out by titrating its solution with strongly ammoniacal copper sulfate solution. The impurities present are not oxidized under the conditions of the analysis, the copper sulfate having a selective action on the sodium formaldehydesulfoxalate or the sodium hyposulfite.

Whereas in the case of the sodium hyposulfite only a small percentage of impurities is usually present, sodium formaldehydesulfoxalate may contain a large percentage of foreign substances—e. g., sodium bisulfite formaldehyde, sodium bisulfite, etc.

Of greater importance than the actual assay of the sulfoxalate is the knowledge of the nature of the impurities present, since the presence of sodium bisulfite formaldehyde seems to be one of the main causes of toxicity in the finished neoarsphenamine, when the former is used in its preparation.

It is indeed fortunate that there is a method which will give the assay of the pure substance, as well as indicate the character and amount of the impurities without resorting to any complicated procedure. All that is necessary is the titration of a sample in three different ways.

[1] Presented before the Division of Industrial and Engineering Chemistry at the 65th meeting of the American Chemical Society, New Haven, Conn., April, 1923, and published in the Journal of the Society.

[2] Heyl and Greer, Am. J. Pharm., **94**, 80 (1922).

[3] Jellinek, "Das Hydrosulfit," Teil 2, p. 100

The author found that the method formerly used for determining the impurities was worthless. Jellinek's directions call for the addition of a large excess of sodium bicarbonatae and vigorous shaking while titrating to a yellow color with 0.1N iodine solution. The results of a series of blank experiments, using only sodium bicarbonate and vigorous shaking while titrating of iodine absorbed was directly proportional to (1) the amount of bicarbonate used, (2) the length of time that the iodine was in contact with the bicarbonate, and (3) the amount of iodine added in excess, and was also dependent on the amount of shaking.

The method which follows is one that will not only check itself but also allows interpretation of the results.

Method

Preparation of Solutions.—Solution 1: Dissolve about 500 grams copper sulfate crystals in 2 liters distilled water; add 1,600 mls. 28 per cent ammonia water and make up to 4 liters with water. This solution is assayed for copper either electrolytically or by the method outlined by Sutton.[4]

Solution 2: A 0.1N sodium thiosulfate solution, standardized by any of the usual methods.

Solution 3: A 0.1N iodine solution checked against a standard sodium thiosulfate solution at each titration. In addition to these solutions an approximately 10 per cent caustic soda solution and 28 per cent (stronger) ammonia water are required.

Procedure.—First Titration.

Notes: (1) The entire procedure must be carried out under anaerobic conditions; the sulfoxalate solution must always be kept under an atmosphere of nitrogen.

(2) Only recently boiled distilled water should be used throughout.

Weigh out 15 grams of sample to be analyzed, put into a 100-ml. volumetric flask, add water, dissolve, and make up to mark. (Call this Solution A.)

Into a 250-ml. volumetric flask place 10 ml. of Solution A and make up to the mark with water. (Solution B.)

Into a wide-mouthed 250-ml. Erlenmeyer flask provided with a 3-hole cork stopper, through one hole of which passes an L-tube reaching to about 1.5 inches above the bottom of the flask, put 50 ml. of Solution 1 and 40 ml. of 28 per cent ammonia water. Warm the mixture to 55 deg. Cent., and keep at this temperature by means of a hot plate or a water bath. While passing in a stream of nitrogen through the L-tube, titrate the copper solution with Solution A until the mixture is colorless. Repeat this titration in order to check the result.

If the percentage of sodium formaldehydesulfoxalate is low, only 30 ml. of ammoniacal copper sulfate solution and 25 ml. of ammonia water are used.

Calculation: 1 ml. ammoniacal copper sulfate solution containing 0.07982 gram copper sulfate is equivalent to 0.0295 gram sodium formaldehydesulfoxalate.

Second Titration: Flush a 250-ml. Erlenmeyer flask with nitrogen. Run into it 25 ml. of the Solution B, and immediately add 0.1N iodine solution until the end-point is reached. Add 2 ml. of iodine solution in excess, allow to stand for five minutes, and titrate back with the 0.1N sodium thiosulfate solution.

Calculation: On the basis of the number of ml. of iodine solution consumed, 1 ml. 0.1N iodine solution is equivalent to 0.00295 gram of sodium formaldehydesulfoxalate.

Note: This result should check very closely with that obtained by the first titration ("Copper" method).

Third Titration: Flush a 250-ml. Erlenmeyer flask with nitrogen. Run into it 25 ml. of Solution B, and immediately add 0.1N iodine solution in amount equal to 50 ml. more than was used in the second titration. Then add 10 per cent sodium hydroxide solution until the color of the mixture is a light yellow. Allow to stand ten minutes. Acidify with dilute hydrochloric acid, and titrate the liberated iodine with 0.1N sodium thiosulfate solution.

Calculation: From the number of milliliters of iodine solution consumed in this titration, subtract one and one-half times the number of millimeters of iodine solution consumed in the second titration. Each millimeter of 0.1N iodine solution in the difference is equivalent to 0.00335 gram of sodium bisulfite formaldehyde.

From the results, we have the percentage of sodium formaldehydesulfoxalate (checked by two methods) and the percentage of sodium bisulfite formaldehyde.

Analysis of Sulfoxalates

A number of sulfoxalates were analyzed and the results were tabulated as follows:

SODIUM FORMALDEHYDESULFOXALATE

Sample	Copper Method	Iodine Method	Sodium Bisulfite Formaldehyde
5320.....	72.7	83.5	....
5368.....	31.4	31.4	52.35
5367.....	75.6	75.9	7.25
5554.....	17.4	17.7	66.0
5584.....	26.2	26.35	61.25
5575.....	23.8	23.95	59.9

The discrepancy in the first sample between the percentages for sulfoxalate obtained by the copper and the iodine methods is only explained by the presence of an impurity other than sodium bisulfite formaldehyde. This impurity must be sodium hyposulfite, for if we assume that any other impurity—e. g., sodium bisulfite—is present, the total of the percentage is above 100 per cent. Then, again, sodium hyposulfite

[4] "Volumetric Analysis," 9th ed., p. 188.

could easily have been formed if insufficient formaldehyde had originally been added, or if much had been lost by evaporation.

The calculation is as follows:

$$Na_2S_2O_4 + 2CuSO_4 + 4NH_4OH = Na_2SO_3 + Cu_2SO_4 + (NH_4)_2SO_3 + (NH_4)_2SO_4 + 2H_2O$$

$$NaHSO_2.CH_2O + CuSO_4 + 3NH_4OH = Na(NH_4)SO_3 + CH_2O + Cu_2SO_4 + (NH_4)_2SO_4 = 2H_2O$$

Thus:

1 $CuSO_4$ = ½ hyposulfite or ½ sulfoxalate or 1 norm $CuSO_4$ = ¼ mol hyposulfite or ¼ mol sulfoxalate.

For the iodine titration:

$$Na_2S_2O_4 + 6I + 4H_2O = 2NaHSO_4 + 6HI$$

$$NaHSO_2.CH_2O + 4I + 2H_2O = NaHSO_4 + CH_2O + 4HI$$

Thus:

1 mol iodine = 1/6 mol hyposulfite or ¼ mol sulfoxalate. Then if x = grams of sodium hyposulfite in the entire solution and y = grams of sodium formaldehyde hyposulfite in the entire solution

$$\text{For the copper titration } \frac{x}{43.5} + \frac{y}{29.5} = a$$

$$\text{For the iodine titration } \frac{x}{29} + \frac{y}{29.5} = b$$

where

a = Norms of copper sulfate used in titration
and b = Norms of iodine used in titration.

Thus, for Sample 5320:

Sodium hyposulfite	30.6 per cent
Sodium formaldehydesulfoxalate	52.0 per cent
	82.6 per cent

ACKNOWLEDGMENT

This work was done in the research laboratory of E. R. Squibb & Sons, during the early part of 1922, while making an investigation of neoarsphenamine and its manufacture.

Finishing Light Broadcloth

Processes Employed—Tacking the Pieces—Washing and Fulling—Control of Shrinkage—Raising the Fibers —Dry Finishing

A SOFT, smooth and firm feel and a lustrous finish are generally wanted on broadcloths for women's wear. In detail the processes employed to get these qualities and produce a successful broadcloth finish are as follows:

The first process after weaving is burling, which should be well and carefully done. If threads have been broken by the burlers, the sewer or mender, who is the next worker to handle the goods, should report this fault, and also repair the damage before passing the cloth along. After burling and mending, or sewing as it is called, has been accomplished, the goods should be carefully inspected, and if any imperfections have escaped the sewer's attention the piece should be returned marked with chalk for her benefit.

Tacking the Pieces

Four pieces which have been passed by the inspector as mended and as free from weaver's and burling imperfections as can be attained are now made up into a set and taken to the tacking machine, where they are tacked with the face inside. This is a simple operation, but it should be performed by one who is expert in the use of the machine; for if the tacking is not kept on the listing and as close to the outside of the listing as the machine will sew, it will not be able to keep the listings from doubling over and felting down. This will cause endless trouble on the different machines used in the finishing.

The gig will work harder on the doubled-over listings, thus making them tender, and if they survive the gigging the shears will destroy them by cutting them up, and a good many times the shears will cut into the cloth as well. Many, many seconds have been made in this way. It will be seen that the tacking is not a minor and unimportant operation, as many consider it.

Another reason for tacking with the face inside is that all the rub caused by friction in washers and fulling mills is confined to the back of the cloth, thus saving the face from unnecessary contact.

Washing and Fulling

The four pieces are taken to the washer and given a soaping with a thin-boiled palm oil or green olive soap. This will remove the grease and dirt from the goods and at the same time impart a softer feel to the cloth. Run about ten minutes in warm water, 120 deg. Fahr., before putting on any soap. Then drain off and add soap, about two pailfuls to each piece, or enough to lather. Run twenty minutes and then turn on the warm water as before for thirty minutes. Now turn off the warm water and run the cold water for five or ten minutes, or until the cloth is cooled off. Next run the cloth out of the washer on to a truck and take to

the extractor; extract more than if the goods were to be dried.

After washing, put the goods into the fulling mills, marking off one yard near the ends of the pieces with white string. This is to guide the fuller in giving the shrinkage necessary to bring the goods to the required finished weight. Thus, if the goods are required to shrink 3 inches per yard, the strings that were put in a yard apart when the goods were started in the mill will be only 33 inches apart when they are fulled enough in length.

Soap the goods up slowly and as evenly as possible with a heavy bodied palm oil soap, using ammoniated potash for a lye. The damp condition of the goods when put into the fulling mills helps greatly in spreading the soap evenly. When enough soap has been run on to be easily lifted with an upward turn of the thumbnail, the goods are well soaped. The writer's rule is to give as much soap as possible, but not enough so that the goods will slip or lather.

If slipping occurs run in another white piece on either side. If another piece is not available for this purpose, take the pieces out and extract a little, then put back into the mills and full slowly, keeping the top doors open. If there is a draft from windows or doors, the covers of the mill near the draft should be closed. A draft is bad for fulling; it dries up the soap and makes the goods chafe and waste, losing weight and felt. It also gives the cloth a dry feel when finished, and a dingy, lusterless look.

Control of Shrinkage

Another point in milling goods is to bring the length shrinkage and width shrinkage up together. If weights are put on the traps to bring the length up in a hurry because the width has come up first, the length is liable to stretch out again. Where the length and width are brought up together, the shrinkage will resist the strain given the cloth in the subsequent operations.

The four pieces, now fulled in length and width, are taken to the washer, run in warm water as before, and lathered well for twenty minutes. Rinse with warm water for twenty minutes. Then drain off and add four pailfuls of the thin soap first used in the scouring, and lather. This lather will be snow-white. Give twenty minutes, then rinse with warm water until the soap is thoroughly washed out. Turn on cold water for fifteen minutes. Take out, extract again, and after pulling out the tacking fold out and sew together end to end.

Then take to rolling and stretching machines. With water in the bowl at 150 deg. Fahr., run on rolls tightly and let stand on end for a few hours. Turn rolls when necessary to keep moisture even. The goods should then be unrolled and wet up by running through a crab or any other machine that will permit them to be wet up in a full width condition.

Raising the Fibers

The hot rolling and stretching having removed all wrinkles and crimps, it is an advantage to keep the cloth that way. After wetting out, run the pieces over a vacuum extractor, and then take to the gigs. Two pieces are put on each teasel gig. Start with old work for one hour, then clean off and, with each cylinder half filled with a grade of better work, give one hour more. Clean off again and put in one-fourth of each cylinder shap work or new teasels; run one hour.

The goods now feel soft and velvety, yet firm. This is the feel we aim to attain. As to looks, the face must appear smooth and even. If any pimpled or plucked appearance still remains, the gigging should be continued until this is overcome. It is sometimes necessary to take the cloth off and turn end for end, running the last end first.

After this process take the goods to the cropping shear. See that this machine is in good order and will cut wet tissue paper. Give a few runs, cutting off and laying the pipe nicely. This means only trimming the nap. The goods are then taken to the wet steamers and four pieces wound on the cylinder tightly, and wrapped and tied snugly. Cold water is turned out until it comes through, then shut off and steam turned on. Ten minutes of steam is given. Now cool off again, unwrap and run on opposite cylinder, repeating the treatment.

The goods are then extracted, once more tacked and sent to the dyehouse. The tacking at this stage is to prevent the goods from rolling up and shading, which invariably happens when goods roll up in the dye kettles.

After coloring, the pieces are returned to the wet finishing room, where they are put into the washer, and after ten minutes of cold water they are given a bath of fuller's earth, rinsed off and taken to the wet gig, where they receive one hour of wet gigging. This lays the pile and shows the lustering to better advantage. Extract again on the vacuum extractor and dry.

Dry Finishing

The goods are now given a few runs on the shear, lightly pressed and sent to be specked. The pressing shows up any specks clearer than if not pressed, and the specker in consequence can do better and more perfect work.

After specking, the goods are again returned to the dry finishing room and given a good steam brushing, after which they are sent to the finishing shears and the pile cut slowly to its exact length. The goods are again pressed and given a final examination before being cased up for the market.—Textile World.

Sulphur and Mordant Dyes

The Katigene Dyes and Their Use—Method for Fast Colors on Silk—Special Use of Sulphur Colors—Mordant Dyes—Chromium Mordant—Mordant Reds—Iron as a Silk Mordant—Silk Fiber for Pile Goods

By GEORGE EMMONS

SILK dyers have apparently been slow to use the celebrated dyes belonging to the sulphur colors. Where the situation is such as to permit of the employment of these dyestuffs, we can pretty well count on considerable to high degrees of fastness against light, water, ammonia, washing, boiling and excess dyeing. These are very desirable qualities, and not always readily obtainable by the silk dyer.

However, there are disadvantages to be weighed in the balance or overcome as best we can. First and foremost of the disadvantages consists in the fact that the sulphur colors are usually dissolved in a very considerable quantity of sodium sulphide. The amount required may run up to four times the weight of the sulphur dye. Furthermore, these dyers are often impure, and require—partly for this reason—to be used in large quantities. For example, it may be necessary to employ a weight of dyestuff equal to 10, 15, 20 or 25 per cent of the weight of the silk. It may easily happen that the weight of sodium sulphide exceeds that of the silk itself. Now, sodium sulphide is a powerful alkali, and strong alkalies act injuriously on the silk fiber. This fact is known, and for a long time retarded the application of the sulphur dyestuffs in silk dyeing.

Nevertheless, efforts have been put forth to discover some substance which would nullify more or less perfectly the bad effects of sodium sulphide. For example, additions of gelatine, tannin, lactic acid and sodium lactate have all been tried or considered. It is admitted that such agents ameliorate the action of sodium sulphide. Apparently, however, some view the matter in such light that they do not allow that complete neutralization of the sodium sulphide is effected. Thus one experienced teacher says that he and his pupils have tried all the methods proposed but have never taken the silk from the dye bath in an uninjured state.

However, others have probably been better satisfied with results. At any rate, the silk dyer may do well to inform himself as to this class of dyestuffs and see whether they may not prove useful under certain conditions. The properties as to fastness are worth going after, unless they can be produced in equal degrees by other dyes. Certain kinds of fastness may, under certain circumstances, be so desirable that the dyer will be willing to accept some of the injurious effects.

The Katigene Dyes and Their Use

The Katigene dyes may be used with silk in accordance with the following directions: These dyestuffs, crystallized with sodium sulphide, are to be dissolved in the usual way. The solution is added to the body of water constituting the foundation of the dye bath. Next we are to add common salt in an amount within the range 20 to 40 per cent, the percentage being based on the weight of the silk. After the salt has been well dissolved, sodium acid lactate in an amount equal to one and one-half times the weight of the sodium sulphide is to be added, a little at a time, and very thoroughly dissolved in and distributed through the dye bath.

If the black dye, Katigene Black T3B, is used for the purpose of producing a black, we are to use more of the sodium acid lactate. That is to say, we use from two and one-half times the weight of the sodium sulphide. This is perhaps the best of the blacks. A little Turkey red oil or monopol soap may be added, together with gelatine. The amount of gelatine is regulated by the size of the dye bath. For each 100 U. S. gallons of liquor, 5 5/6 ounces of gelatine are to be used. The sodium acid lactate is to neutralize the sodium sulphide in respect to injurious activity, and it serves to cause the bath to draw better.

The dyeings of colored shades selected from this group of dyestuffs is to be carried out preferably at 50 or 60 deg. Cent. (122 or 140 deg. Fahr.). The blacks are to be dyed at a higher temperature; say, in the range 80 to 90 deg. Cent. (176 to 194 deg. Fahr.). The dyeing lasts for one hour, during which period the silk is kept submerged. Iron rods are suitable for supporting the silk. No copper is to be permitted anywhere.

Another method may be used with the Katigene dyes. That is to say, caustic soda and sodium hydrosulphite are used, and the bath is a long one, the ratio of silk to water by weight being 1 to 40.

If it is desired to use a Katigene dye producing a colored effect, we may use of the dyestuff 15 per cent of the weight of the silk. With this we employ from 3 to 5 c.c. of caustic soda solution having a density corresponding to 30 deg. Be., from 1½ to 2½ grams of hydrosulphite (concentrated powder, BASF), and from 30 to 40 grams of common salt to each liter of bath liquor.

For a black we may use 40 per cent of the Katigene dye, the percentage being based on the weight of the silk. For each liter of the bath liquor, we add 13 to 15 c.c. of caustic soda, from 5 to 6 grams of hydrosulphite, and 50 grams of common salt. The dye is made into a paste along with the caustic soda. That is, to these two substances we add and mingle warm water, leaving a temperature of about 60 deg. Cent. (140 deg. Fahr.). When a paste has been made, the hydrosulphite powder is then strewn into the mass and the whole left alone until the dye has dissolved. We now have a concentrated dye solution. Before adding it to the body of water constituting the foundation of the dye bath, we dissolve in this water the common salt. The concentrated dye solution is then added.

The silk is dyed for one hour at 60 deg. Cent. (140 deg. Fahr.). It is preferably kept submerged during the whole of the period. It is next rinsed and hung up for a short time. After this, the silk is put through a souring bath containing acetic or formic acid. This is followed by a rinsing and then a brightening with formic or acetic acid. The second method of dyeing described is also suited for dyeing with the hydron dyes. These may be viewed as dyestuffs standing between the sulphur dyes and the vat dyes.

Some may think that all this about sulphur dyes and silk has but little real, practical importance. That may depend upon the situation. These dyestuffs are probably not first-rate, all-around dyes for silk fiber. At the same time, they have importance because of their suitability to meet certain special conditions. Let me quote from leading experts ("Manual of Dyeing," Knecht, Rawson and Loewenthal): "But silk is well dyed with these dyestuffs if required to be fast to light, washing, bleeding in water, boiling-off, acids, etc."

Method for Fast Colors on Silk

Here is a method which is understood to be adapted to the production of fast colors on silk (see English Patent 24697, 1901): The sulphur dye in proper amount, the suited proportion of sodium sulphide, and a weight of grape sugar equal to twice the weight of the sodium sulphide are all boiled together in a quantity of water for ten minutes. This solution is to be considered the concentrated dye solution.

While it is being prepared, or prior to its preparation, the main dye bath may be gotten ready up to a certain point. That is, the plain water for the bath is filled into the tub or tank. To this water—that is, to every 12 U. S. gallons of it—we add 1½ ounces soda ash, 5 ounces Turkey red oil and ½ to 1 ounce of Glauber salt. This bath is to be used boiling hot. The concentrated dye solution is added and the silk yarn dyed for one hour. If rods are used, they are to be in the form of bent rods. This makes it easy to submerge and keep submerged the fiber that is being dyed. After dyeing, the silk is lightly pressed off the rods and then given the rinsing and soaping baths. Afterward the yarn may be brightened in a brightening bath based upon acetic acid.

In case the colors thus dyed upon the silk are desired to be fast against boiling-off in soap liquor, we may apply the following as an after-treatment. That is, a boiling-hot bath is prepared by making additions to plain water of 2 per cent of the weight of the fiber in the form of copper sulphate, 2 per cent of bichromate of potash, and 5 per cent of acetic acid. The work is next rinsed, and then brightened. The addition of the grape sugar is for the purpose of neutralizing the injurious action of the sodium sulphide. Similar effects are produced by tannin, formaldehyde, acids, acid salts; and ammonium salts also serve this purpose to some extent.

If black is to be applied, we may advantageously first employ a mordanting liquor containing nitrate of iron. This liquor may have a density corresponding to the specific gravity 1.125 to 1.160 (25 to 32 deg. Tw.).

Special Use of Sulphur Colors

Certain of the sulphur colors may be utilized in a special manner when it is desired to prevent the silk fiber in a mixture from absorbing any of the dyestuff. This prevention is secured by adding glue to the dye bath. In this way non-silk fiber may be suitably dyed and the silk threads used for decorative purposes left undyed. The non-silk fiber will probably be cotton, as the sulphur colors are not especially well adapted for dyeing wool. We may dye the cotton black or some fancy color and leave the silk threads untouched. They may be white from bleaching or may have their own special fancy color, having been dyed prior to weaving.

Silk-cotton may be dyed by the process already described, which calls for the use of grape sugar. It has been recommended to use additions of sodium phosphate, sodium borate or sodium silicate for the purpose of protecting the silk against the injurious action of the sodium sulphide.

This same procedure—that is, dyeing with the addition of grape sugar—may be employed in dyeing ordinary satins. Black satins that have been produced by dyeing with some sulphur dye may be suitably topped with logwood. Presumably this is to give luster and perhaps add to the opaqueness.

Mordant Dyes

Silk may be successfully dyed with mordant dyes. The situation here is similar to that created by the proposal to use sulphur colors. While the mordant dyes are applicable, they are not to be considered the class of dyestuffs to use upon general occasions. They are to be resorted to when some special result is wanted, and it is permissible to pay for this desired result in money and perhaps in some sacrifice of beauty. In general, the acid and

the basic dyes may be fixed on silk at less expense than the mordant colors. But, if very special results in fastness are required, we have to turn to some such processes as that which makes use of the mordant dyestuffs. We should expect some reduction in luster and pliability.

Aluminum.
Chromium.
Iron.

In general, when the mordant dyes are applied properly and the most suitable of these mordanting metals is chosen, we may expect the final result to be very fast against light, soap or milling. The colors are much faster on silk than the acid or basic aniline colors.

In making up the dye bath, we may use 80 per cent water and 20 per cent boiled-off liquor. No soda should be present in the boiled-off liquor that is employed. There should be present only a moderate proportion of silk gum. This means that the boiled-off liquor should be neither too old nor too slimy. The liquor remaining after the second boiling-off will probably be just about the right thing. The size of the dye bath calls for attention. It should weigh around 25 times the weight of the silk. We may go up to 30 and down to 20. In order to avoid weighing the water, we may use the fact that 8 1/2 pounds of water amount to one U. S. gallon, and that 10 pounds of water amount to one British imperial gallon. If the silk weighs 100 pounds, then we are to use, say, 2,500 pounds of water. This amounts to 300 U. S. gallons or 250 British gallons.

Aluminum will often be used in the form of alum. It is indicated when lighter and brighter shades are wanted. If darker shades are desired, then chromium may seem preferable. Where iron is to be employed, we may usually turn to basic nitrate of iron. Chromium is to be used in the form of normal or basic chromium salts. The bichromate of potash is ordinarily not to be used for mordanting purposes with silk. It is, nevertheless, employed as an oxidizing agent in the development of Cutch browns.

The basic-alum mordanting bath for silk may be prepared by using the following recipe:

Alum	10 parts
Soda (crystals)	1 part
Water	As directed
Tin crystals	3/64 part

The first three ingredients are dissolved in sufficient water to bring about complete solution. This may mean 150 parts. All parts are to be understood as parts by weight. The liquor is to be gently heated until a precipitate which forms at the beginning has completely disappeared. Or, instead of alum, we may employ sulphate-acetate and nitrate-acetate in a similar manner.

Before introducing the silk into this mordanting liquor, we thoroughly wet it out and then wring as evenly as possible. The even wringing is required, apparently, in order to get even results in the mordanting bath and in the dye bath. After the wringing is done, the silk is transferred to the mordanting liquor, where it is turned for a short period without any application of heat. After the turning, the silk is allowed to soak for, say, twelve hours. It may now be wrung and washed in water; or preferably it may be passed in advance through a solution of sodium silicate having a density corresponding to the specific gravity 1.005 (1 deg. Tw.). The purpose of this treatment is to fix the alumina. But, instead of the sodium silicate liquor, we may employ a soap bath. Finally, the silk is thoroughly washed for the purpose of getting rid of all surplus chemicals. Next it goes, without drying, into the dye bath.

In using chromium, we may employ with ungummed silk, for example, a strong solution of basic chromium chloride. This liquor may have a density corresponding to 1.160 specific gravity (32 deg. Tw.). The ungummed silk is steeped in it for, say six hours, when it is to be well wrung out, washed, and then worked for fifteen minutes in a solution of sodium silicate having the density corresponding to 1.0003 specific gravity (½ deg. Tw.).

Chromium Mordant

For discharged silk, we may employ Chromium Mordant GA, III (M.L.B.) for dyeing alizarin colors. The discharged silk is to be steeped for a long period, say twelve to twenty-four hours, in a solution of the mordant. This solution may be prepared by dissolving the mordant in four parts of water. The silk is next washed with water, when it will be ready for the dyeing.

When iron is to be used to mordant silk, we may ordinarily make use of the so-called nitrate of iron, although it does not contain either ferric nitrate or ferrous nitrate. Permit me to give as a means of identification of the basic nitrate of iron the following formula:

$$Fe_4(SO^4)_5(OH)_2$$

We should be careful to obtain, or make, the right thing. In general, it is understood that the brands of basic nitrate of iron made for cotton are not quite basic enough.

When preparing the dye bath for use after mordanting with aluminum or chromium, we neutralize the alkaline character by adding acetic acid for aluminum, and make the bath slightly acid for chromium.

As a rule the azo reds have strong tinctorial power, and a 2 per cent amount o fthe dyestuff will be sufficient to produce a full shade. This percentage is based on the weight of the fiber. Pale tints may be produced advantageously with such small amounts as one-eighth or one-fourth of one per cent.

Alizarine is the chief representative of this class. It is one of the older artificial coal-tar colors. Mordant colors are not entirely successful in silk dyeing, as the fiber has

hardly sufficient affinity for some of the best mordants; but with careful attention very good results can be got with Alizarine. There are several procedures.

Method No. 1.—After the silk has been suitably scoured, it is treated in a mordanting liquor prepared in accordance with the following recipe:

Alum	45 %
Soda crystals	4.7%
Water	750 %

These percentages are based on the weight of the silk. The water required weighs thus seven and one-half times as much as the fiber. This bath is to be boiled until the precipitate which forms at the start has been thoroughly redissolved. The silk is then entered and worked for fifteen to twenty minutes. It is then permitted to remain for a long period—say twelve hours. It is next wrung out and put through the centrifugal machine. Complete dryness is not necessary, so that the silk coming from this machine will be in proper condition. The alum bath may be preserved and used again, if it is properly freshened by the addition of alum and soda.

The next operation to be applied to the silk consists in a treatment in a bath containing sodium silicate. This bath is to contain enough of this aricle to give a hydrometer reading of 1 deg. Tw. (1.005 specific gravity). The silk is entered into this bath and worked for twenty or twenty-five minutes. The hanks are then lifted, wrung and thoroughly washed and then wrung again. The silk is now ready to go into the dye bath.

This may be prepared in accordance with the following recipe:

Alizarine (20 per cent)	As directed
Water	1,000 pounds
Boiled-off liquor	24 U. S. gallons

In contemplation here are ten pounds of silk. The water requirement may be reduced to U. S. gallons by dividing by 8 1/3.

It is considered necessary to be quite particular as to the boiled-off liquor specified in the recipe. It must be free from soda. Besides, it must not be too strong in silk glue. It is recommended that the liquor from a second boiling off be used, or even a fresh, neutral soap liquor. In any case, weak acetic acid is to be added to the bath until the latter is quite neutral.

The bath is made up by adding to the plain water the boiled-off liquor and following this with the dyestuff. If the red wanted is a medium shade, we use one or two pounds of the 20 per cent alizarine; if a deep shade, from two to five pounds. If merely a pink is desired, we may cut the amount to one-half or one pound. After the silk has been duly entered in the bath, it is worked for fifteen to twenty minutes, the bath being left cold. Then heat is turned on, a little at a time, so that the boiling point will be reached in the space of fifty-five minutes or longer. It is said that rapid heating results in loose color. The boiling point, once reached, is maintained for one hour, after which the silk is washed.

In order to brighten the color, which will doubtless have dulled because of the sericin, etc., present in the dye bath, we may proceed as follows: That is, we may prepare a weak soap bath by dissolving two ounces of soap in sixty pounds of plain water and boil the silk in it for twenty minutes. The silk is now removed and then given a good washing. A second bath will have been prepared in the meantime by adding two and one-half to three pounds acetic acid, 10 deg. Tw., to every 100 pounds of water. This bath is used at a temperature of 100 to 120 deg. Fahr. The silk is worked for ten or fifteen minutes, wrung and then dried.

Iron as a Silk Mordant

We have already had before us the use of chromium of alumin as mordanting metals. It is now in place to say a word or two as to iron. One of the most important applications of iron to silk has in view the weighting as well as the dyeing of the fiber. Black is the color in contemplation here.

The weighting of black silk is too large a matter for attention just now. However, ferrous sulphate—an important iron salt—is employed in connection with the production of the dyed effect known as English Black. No weighting needs to be in contemplation in this connection.

English Black is applied to tender silk materials which do not permit many treatments or which are to be interwoven with white.

The following recipe is suitable for silk yarn:

Logwood	50%
Fustic	50%
Ferrous sulphate	5% to 6%
Copper acetate	2% to 3%

The percentages here are based on the weight of the dry silk yarn. We prepare the dye bath in accordance with the foregoing schedule of ingredients and then warm it to 50 or 60 deg. Cent. (122 or 140 deg. Fahr.). The silk yarn is then entered and dyed for a period of thirty minutes. The yarn is next lifted free of the bath, and the latter heated up to 70 deg. Cent. (158 deg. Fahr.), when the silk is again put in and the dyeing prosecuted for a second period of thirty minutes. While the hanks are still hanging on the rods, the dye liquor is run off and the silk left in the empty beck for, say, one hour. At the expiration of this period, the silk is given a good rinsing.

We now use a second dye bath, prepared in accordance with the following recipe:

Logwood	50%
Soap	50%

As before, these percentages refer to the silk as the basic 100 per cent. This new bath is warmed to 50 or 60 deg. Cent. (122 or 140 deg. Fahr.) and the silk entered. During a dye period of half an hour, the hanks are continually turned to equalize the coloring effect. Then the silk is lifted clear of the bath and the temperature pushed up to 70 or 75 deg. Cent. (158 or 167 deg. Fahr.), when the silk is re-entered and then turned for a second half-hour. While the silk is still in the beck, the dye liquor is run off and the fiber rinsed where it is. Afterwards, a second rinsing is given in running water. This operation is succeeded by the removal of the water in a centrifugal machine. The yarn may now be brightened and softened by means of 2 to 4 per cent of oil which has been mingled with just enough acid to give a sour taste.

If the foregoing operations are well and successfully carried out, we are to expect a fine black with a greenish tone. No weighting is to be expected.

Silk Fiber for Pile Goods

Silk fiber destined for pile goods may be dyed without weighting in the following manner: The mordants here are iron and aluminum. The first thing to do is to work the goods for a period in a cold, concentrated solution of alum, and then allow them to steep in the liquor for, say, twelve hours. At the expiration of this period, dyeing is to be carried out as if for English Black, only now we omit the fustic and dye in a bath containing 50 per cent of logwood, 3 or 4 per cent of copper acetate and a small amount of ferrous sulphate. The second dye bath is to be a logwood bath containing soap. When using the ferrous sulphate in the first bath, we employ a less amount than when seeking to produce English Black on yarn. The softening is effected in a tepid bath of a weak character. This is a liquor made with neutral soap. After the softening, the silk yarn is put through the centrifugal machine and then dried. No rinsing is permitted. It is undesirable to luster the yarns while they are moist, because of the excessive deepening of the shade.

Prior to the mordanting operation with the alum, it is permissible to dye the silk with Methyl Violet or Aniline Blue, the purpose being to give a fine, bluish tone to the finished work. However, there is more or less danger of stripping the color from the silk during the succeeding operations. Carefulness as to this point is accordingly to be exercised. No weighting is to be expected.

Black velvets and plushes produced by this procedure may be expected to disclose a blue-black when viewed direct (underhand) and a black-blue when viewed through (overhand). Aluminum and iron are both used here, as may be seen from the directions.—Silk.

A call for motion picture films to be shown at the 1923 Chemical Exposition when it is held this fall at the Grand Central Palace, New York, during the week of September 17 to 22, has been sent by the management to exhibitors.

PARA RED COLORS ON COTTON GOODS

(*Continued from page 584.*)

In a dyeing plant where it is not possible to treat the yarn prepared with the beta-naphthol at once with the diazotized para bath, an alkaline antimony oxide solution containing glycerine is introduced in the mordanting bath somewhat as follows:

- 27 parts beta-naphthol are dissolved warm in
- 27 parts caustic soda lye at 40 deg. Be. and to the clear solution is added
- 100 parts castor oil soap and
- 1,000 parts water. The clear solution thus formed is mixed with
- 200 parts water containers.
- 4 parts antimony sulphate made clearly soluble with caustic soda and glycerine.

The above, with the addition sometimes of tartaric acid, is also the grounding used for para red for printing and with the addition of sodium acetate for Diamsidin Blue.

The following development bath is practised by one of the most successful para red dyers. The mixtures are made in two parts as follows:

Part 1.—Two pounds 13 ounces Paranitraniline C are mixed with 2 gallons of boiling water and dissolved by the addition of 4½ pints of hydrochloric acid, 36 deg. Tw. This solution is stirred into about 5 gallons of cold water and cooled to 100 deg. Fahr., by the addition of 10 pounds of ice; 1 gallon 4½ pints of a solution of nitrite of sodium (1:10) are then added. After a few minutes the solution will have become clear, when it is diluted with cold water to 15 gallons.

Part 2.—Six pounds sodium acetate are dissolved in 4 gallons water and diluted with water to 5 gallons.

The developing bath is composed of parts of Part 1 and one part of Part 2.

In dyeing paranitraniline red, it may be laid down as a rule that in order to get a uniform bright red with a bluish cast, the first essential points are that the beta-naphthol preparation must contain 5 to 10 per cent of the beta-naphthol, together with a slight excess of caustic soda over that amount necessary to form the normal naphtholate of soda and an addition of the ammonia soap of castor oil; for instance:

- 4½ pounds beta-naphthol.
- 6½ ounces Red Developer C (Casella).
- 5 pounds caustic soda lye (75 deg. Tw).
- 6½ pounds boiling water; then add
- 10 pounds castor oil soap dissolved in 5 gallons of hot water, then dilute the whole to 20 gallons with cold water; dry, and develop as quickly as possible.

In the calico printing business paranitraniline red can be applied to cloth in the following variety of ways:

1. By padding the cloth in beta-naphthol printing paste, and then developing it into a solid red in a diazo solution.

2. By coupling diazo compounds with certain other compounds containing free adimo groups.

3. By printing a pattern in a thickened solution of naphthol and afterwards developing in a diazotizing bath.

4. By printing a thickened diazo solution on cloth prepared with beta-naphthol.

5. By printing on cloth prepared with beta-naphthol certain reducing agents capable of reducing the diazo compounds before they can unite with the beta-naphthol.

6. By discharging a red ground (dyed by Method 1) with reducing agents acting under the influence of hot dry steam.

The important feature indicated by the above is the rather unusual fact that paranitraniline red is capable of being either reduced or discharged by the same agent, which is quite a convenience.—Textile American.

Foreign Dyes Imported Through the Port of New York During July

Including Synthetic Chemicals, Medicinals, Photographic Chemicals, Intermediates, and Other Coal-Tar Products Classified by the Tariff Act of 1922 Under Paragraphs 27 and 28

Compiled by the Chemical Division of the Bureau of Foreign and Domestic Commerce, in Collaboration with the Chemical Division of the United States Tariff Commission

KEY TO ABBREVIATIONS FOR FOREIGN MANUFACTURERS

1—The Six Leading German Companies

A—Actien-Gesellschaft fur Anilin-Fabrikation, Berlin. Founded 1873. Branches in France and Russia.

B—Badische Anilin-und Soda-Fabrik, Ludwigshafen on the Rhine. Founded 1865. Branches in France and Russia.

By—Farbenfabriken vorm. Friedr. Bayer & Co., Leverkusen on the Rhine. Founded 1862. Branches in France and Russia.

C—Leopold Cassella & Co., Frankfort on the Main. Founded 1870. Branches in France and Russia.

K—Kalle & Co., A. G., Biebrich on the Rhine. Founded 1870. Branches in Russia.

M—Farbwerke vorm. Meister Lucius & Bruning, Hochst on the Main. Founded 1862. Branches in France and Russia.

2—The Seven Smaller German Companies

BK—Leipziger Anilinfabrik Beyer & Kegel, Furstenberg near Leipzig. Founded 1882.

GG—Chemikalienwerk Greishem G.m.b.H., Griesheim on the Main. Founded 1881.

CJ—Carl Jager G.m.b.H., Anilinfarbenfabrik, Dusseldorf. Founded 1823.

GrE—Chemische Fabrik Griesheim-Electron, Offenbach on the Main. Founded 1842.

L—Farbwerk Muhlheim vorm. A. Leonhardt & Co., Muhlheim on the Main. Founded 1879. Branch in France.

tM—Chemische Fabriken vorm. Weiler ter Meer, Uerdingen on the Rhine. Founded 1877.

WD—Wulfing, Dahl & Co., A. G. Barmen. Founded 1842.

3—Dutch, Belgian and French Companies

FA—Farbwerk Ammersfoort, Ammersfoort, Netherlands. Founded 1888.

NF—Niederlandische Farbe-und Chemikalienfabrik Delft, Netherlands (Delft). Founded 1897. Branch in Russia.

LG—Lazard Godchaux, of Brussels. (These products are probably compounded largely from the dyes made by A. Wiescher & Co., of Haeren, Belgium.)

P—Societe Anonyme des Matieres colorantes et produits chimiques St. Denis (formerly A. Poirrier), St. Denis, near Paris, France. Founded 1830.

4—Swiss Companies (all at Basel)

DH—Farbwerke vorm. L. Durand, Huguenin & Co. Founded 1871. Branches in Germany and France.

G—Aniiinfarben-und Extract-Fabriken vorm. Joh. Rud. Geigy. Founded 1764. Branches in France, Germany and Russia.

I—Geselfschaft fur chemische Industrie. Founded 1885. Branch in France.

S—Chemische Fabrik vorm. Sandoz & Co. Founded 1887.

5—English Companies

BAC—The British Alizarin Co., Ltd. Established 1882.

ClCo—The Clayton Aniline Co., Ltd., Clayton, near Manchester Founded 1876.

CR—Clauss & Co. (formerly Clauss & Ree), Clayton, near Manchester. Founded 1890.

CV—Colne Vale Chemical Co., Milnsbridge, near Huddersfield.

RHS—Read Holliday & Sons, Ltd., Huddersfield. Founded 1820. (Purchases by British Dyes, Ltd.)

BD—British Dyes, Ltd Founded 1915.

Lev—Levinstein, Ltd., Crumpsall Vale, near Manchester. Founded 1864.

Q—Importations of unknown source, through dealers in colors.

THE imports of coal-tar dyes for July totaled 144,687 pounds, with an invoice value of $142,428. The following table shows the monthly imports through the Port of New York for the year 1923:

Month	Pounds	Value
January*	179,309	$185,344
February	191,709	199,690
March	312,809	301,436
April	242,022	256,751
May	261,869	292,340
June	247,174	257,803

*Not complete.

The five dyes leading in quantity imported were Ciba Bordeaux B, Hydron Pink FF, Sulphoncyanine

Black Trisulphon Brown 2G and Erio Green B. Of the total quantity imported in July, 1923, 46 per cent came from Switzerland, 43 per cent from Germany, 9 per cent from Italy, 1 per cent from England and 1 per cent from other countries.

The imports of synthetic aromatic chemicals for July totaled 5,821 pounds, with an invoice value of $12,208. The imports of medicinals, photographic developers and other coal-tar chemicals for July totaled 122,073 pounds, with an invoice value of $31,748.

The dyes in this report are grouped by Schultz numbers, and in the case of those which could not be identified by Schultz number the classification according to ordinary method of application was adopted. As the pastes and powders of the vat dyes vary widely in strength and quantity, each vat dye has been reduced —in nearly every case—to a single-strength basis.

The designation "†" for competitive and "*" for non-competitive indicates the appraisement basis for the assessment of the ad valorem duty in paragraph 28 of the Tariff Act of 1922. Those dyes without designation are doubtful, pending further investigation. The ad valorem rate for competitive dyes is based on the American selling price, as defined in subdivision (f) of Section 402 of Title IV; the ad valorem rate for non-competitive dyes is based on the United States value as defined in subdivision (d) of Section 402 of Title IV of the Tariff Act of 1922.

German Dye Production for June

In conjunction with the July statistics covering the imports of dyes, the following information, received by cable from Trade Commissioner F. E. Breithut, dated July 25, is rendered relative to the German production of dyes for the month of June according to groups. In addition, the production is rendered according to factories:

Group and Classification	Quantity (pounds) May	June
I—Alizarine Red	253,264	209,035
II—Indigo paste	47,740	576,400
III—Vat colors except Indanthrene Blue GCD	269,526	236,385
IV—Indanthrene Blue GCD	1,434	Zero
V—Alizarine colors other than Red	172,753	177,619
VI—Direct colors for cotton	2,420,202	2,750,097
VII—Direct colors for diazotization	158,418	281,221
VIII—Acid colors for wool	1,405,122	1,305,005
IX—Chrome colors for wool	712,158	709,799
X—Basic colors	300,916	270,257
XI—Sulphur colors	1,567,896	2,098,554
XII—Lake colors	144,663	247,782
XIII—Intermediate products	.....	.....
Total	7,454,092	8,862,154

Factories	(pounds)
Barmen	29,251
Berlin	1,397,317
Beibrich	499,162
Dusseldorf	21,639
Frankfort	1,960,552
Greisheim	715,150
Hoechst	576,400
Leverkusen	3,089,680
Ludwigshafen	131,111
Mulheim	57,578
Uerdingen	384,314
Total	8,862,154

Badische produces only Indanthrene Black blau gelb for printing, and Kryogene Black. Hoechst produced only Indigo paste. The June production represents an increase of 19 per cent over that of May. It is interesting to note the large production increase featured in Indigo paste, sulphur and direct colors for diazotization. This is an indication of the efforts of Germany to regain the Far East market.

Schultz No.	Name of Dye and Manufacturer	Quantity (pounds)
11	Chloramine Orange	1,653
	*Chlorantine Fast Brown RL—(I)	
28	Hansa Yellow G	721
	Hansa Yellow G Paste—(M)	
100	†Eosamine	2,000
	*Eosamine B—(A)	
126	Indoine Blue	10
	Naphtindone BB—(C)	
159	Acid Alizarine Black	882
	*Acid Alizarine Black R—(M)	
161	Fast Red	55
	†Fast Red AVX—(B)	
168	Amaranth	200
	*Amaranth B—(C)	
177	Mordant Yellow	200
	Milling Yellow O—(C)	
220	Palatine Black	2,000
	†Wool Black GRF—(A)	
265	Sulphoncyanine Black †	6,614
	*Acid Milling Black B—(G)	
273	Diaminogene Blue	1,498
	*Diaminogene Blue NA—(C)	
274	Diaminogene	1,050
	Diaminogene Extra C—(C)	
	*Diazo Indigo Blue 2RL—(By)	
279	Benzo Fast Scarlet	22
	†Benzo Fast Scarlet GS—(By)	
283	Bismarck Brown	250
	*Bismarck Brown—(Q)	
319	Diamine Scarlet	250
	*Diamine Scarlet 3B—(C)	
	*Diamine Brilliant Bordeaux R—(C)	

Schultz No.	Name of Dye and Manufacturer	Quantity (pounds)
325	Columbia Blue	10
	Columbia Blue R—(A)	
326	Oxydiamine Violet	383
	*Benzo Violet O—(By)	
332	Benzo Fast Red	55
	†Benzo Fast Red 8BL—(By)	
358	Diphenyl Red	110
	*Chloramine Red 8BS Conc.—(By)	
387	Columbia Blue G	25
	Columbia Blue G—(A)	
392	Toluylene Orange	882
	*Direct Fast Orange K—(I)	
424	Chicago Blue	220
	Brilliant Benzo Blue 6B—(By)	
448	Diamine Bronze	200
	Diamine Bronze G—(C)	
457	Trisulphon Brown 2G	5,004
	Trisulphon Brown 2G Conc.—(S)	
496	Setoglaucine	1,161
	*Setoglaucine 753—(G)	
535	Methyl Alkali Blue	145
	Methyl Alkali Blue Pdr.—(GrE)	
	Methyl Alkali Blue—(M)	
539	Soluble Blue	10
	†Water Blue, large lumps	
543	Patent Blue	2,205
	†Patent Blue V—(M)	
546	Cyanol	1,000
	*Blue Extra—(C)	
559	Victoria Blue B	25
	†Victoria Blue B Base—(B)	
562	Fast Acid Blue	22
	†Brilliant Wool Blue G Extra—(By)	
564	Naphthalene Green	5,000
	Erio Green B Supra—(G)	
571	Rhodamine 6G	830
	*Rhodamine 6G Extra—(I)	
	*Rhodamine 6G Conc.—(G)	
	*Rhodamine 6G Extra—(By)	
	*Rhodamine 6G Extra—(A)	
573	Rhodamine B	1,212
	†Rhodamine B Conc.—(G)	
	†Rhodamine B Extra—(B)	
	†Rhodamine B Extra—(I)	
603	Acridine Orange	1,102
	*Acridine Orange DHE—(DH)	
606	Phosphine	2,407
	*Phosphine 3R—(A)	
	*Coriphosphine OX Extra—(By)	
615	Thioflavine S	25
	†Direct Yellow S—(C)	
618	Thioflavine T	668
	Thioflavine T—(Q)	
	*Basic Yellow TCN—(C)	
	*Rhoduline Yellow 6G—(By)	
636	Prune	110
	Naphtha Chrome Violet 2R—(I)	
661	Thiomine Blue	1,103
	*Thiomine Blue G—(I)	
	*Thiomine Blue GO—(M)	
735	Pyrogene Indigo	882
	†Pyrogene Indigo—(I)	
760	Indan. Golden Orange (sgle. stngth.)	2,110
	*Indan. Golden Orange G Dbl. Pst.—(B)	
	*Helindone Gldn. Or. IG Dbl. Pst.—(M)	
761	Indan. Golden Orange R (sgle. stngth.)	175
	Indan. Golden Orange RRT Pdr.—(B)	
762	Indan. Scarlet GS (sgle. stngth.)	55
	*Indan. Scarlet GS Pdr.—(B)	
765	Indan. Green B (sgle. stngth.)	440
	Indan. Black BB Dbl. Pst.—(B)	
779	Alizarine Orange	20
	†Alizarine Orange Pdr.—(M)	
780	Alizarine Red	1,079
	*Alizarine Red S Pdr.—(By)	
	*Alizarine Red W Pdr.—(By)	
792	Cibanone Orange R (single strength)	1,760
	Cibanone Orange R Powder—(I)	
794	Cibanone Black (single strength)	1,102
	Cibanone Black B Paste—(I)	
800	Anthracene Blue	3,000
	†Anthracene Blue WBV Paste—(B)	
	*Anthracene Blue WGV Paste—(B)	
804	Alizarine Blue S	220
	Alizarine Blue S Powder—(By)	
808	*Alizarine Green S	1,696
	*Alizarine Green S Paste—(BAC)	
812	Indan. Orange RT (single strength)	336
	Vat Orange R—(GrE)	
819	Algol Red RFF (single strength)	1,023
	*Algol Red R Extra Paste—(By)	
	*Algol Red R Extra Powder—(By)	
820	Algol Brilliant Violet R (sgle. stngth)	320
	*Algol Brilliant Violet R Paste—(B)	
827	Indan. Claret B Extra (single strength)	496
	*Indan. Bordeaux B Extra Paste—(B)	
831	Indan. Red BN (single strength)	55
	*Indan. Red BN Extra Paste—(B)	
833	Indan. Olive R (single strength)	500
	*Vat Olive R Paste—(By)	
842	Indan. Blue GCD (single strength)	220
	†Indan. Blue GCD Double Paste—(B)	
849	Indan. Yellow (single strength)	224
	Vat Yellow RG Paste—(GrE)	
855	Alizarine Pure Blue B	469
	*Alizarine Sky Blue B—(By)	
	Alizarine Pure Blue B—(By)	
856	Alizarine Astrol	667
	*Alizarine Astrol B Powder—(By)	
858	Alizarine Saphirol B	660
	†Alizarine Light Blue B Conc.—(S)	
859	Cyananthrol R	33
	*Cyananthrol R—(B)	
	*Cyananthrol RXO—(B)	

Schultz No.	Name of Dye and Manufacturer	Quantity (pounds)
862	Alizarine Blue Black	1,261
	Alizarine Blue Black 3B—(By)	
	Alizarine Blue Black 3B—(M)	
865	Alizarine Direct Green G	229
	†Aliz. Cyanine Green G Ex. Pdr.—(By)	
894	Alizarine Indigo B	1,102
	*Alizarine Indigo Green B Paste—(By)	
895	Alizarine Indigo 3R	462
	†Alizarine Indigo 3R Paste—(By)	
901	Ciba Violet B (single strength)	3,968
	*Ciba Violet B Paste—(I)	
907	Ciba Scarlet (single strength)	2,694
	*Ciba Scarlet G Extra Paste—(I)	
	Helindone Fast Scarlet C Paste—(M)	
	*Thioindigo Scarlet 2G Paste—(K)	
908	Ciba Red R (single strength)	551
	Ciba Red R Paste—(I)	
910	Helindone Pink (single strength)	36
	*Helindone Pink BN Paste—(M)	
913	Helindone Orange R (single strength)	2,180
	*Helindone Orange R Powder—(M)	
915	Helindone Fast Scarlet R (sgle. stngth)	2,740
	*Helindone Fast Scarlet R Powder—(M)	
919	Ciba Bordeaux B (single strength)	22,601
	*Ciba Bordeaux B Powder—(I)	
	*Ciba Bordeaux B Paste—(I)	
922	Aniline Black	100
	*Diphenyd Black Base I—(M)	
923	Ursol	50
	*Fur Gray G—(A)	

UNIDENTIFIED ACID DYES

Name of Dye and Manufacturer	Quantity (pounds)
*Alizarine Rubinol R Powder—(By)	471
*Alizarine Uranol R Powder—(By)	220
*Brilliant Acid Blue EG Conc.—(By)	500
*Brilliant Milling Blue B—(C)	10
Neolan Blue 2G—(I)	220
Neolan Gray B (new)—(I)	110
Neolan Rose B—(I)	551
*Polar Yellow 2G Conc.—(G)	2,059
Supramine Black BR	463

UNIDENTIFIED VAT DYES

Name of Dye and Manufacturer	Quantity (pounds)
*Algol Brown G Paste—(By)	1,022
Ciba Pink (single strength)	2,477
*Ciba Pink BG Paste—(I)	
*Ciba Pink BG Powder—(I)	
*Helindone Golden Orange IRRT Paste—(M)	968
†Helindone Pink R Extra Paste—(M)	951
†Helindone Yellow DIG Double Paste (single strength)—(M)	2,000
Hydron Brown (single strength)	1,000
*Hydron Brown C Paste—(C)	
*Hydron Brown R Paste—(C)	
*Hydron Orange R Paste—(C)	110
†Hydron Pink FF Paste	20,331
Hydron Violet (single strength)	495
Hydron Violet R Paste—(C)	
Hydron Violet R Powder—(C)	
*Indanthrene Violet BN Extra Paste—(B)	449
Indigo Natural (50 per cent pure Indigo)	900

UNIDENTIFIED MORDANT AND CHROME DYES

Name of Dye and Manufacturer	Quantity (pounds)
*Acid Alizarine Gray G—(M)	100
†Anthracene Chromate Brown EB—(C)	1,000
Brilliant Chrome Blue 2B—(DH)	110
*Chloramine Light Blue BS—(S)	200
Chromanol Black RVI—(DH)	55
*Chromanol Blue NR—(DH)	110
Cloth Yellow R Powder—(GrE)	100
*Chromorhodine BN—(DH)	220
*Chromoxane Brilliant Blue G—(By)	661
†Eriochrome Flavine A Conc.—(G)	1,914
*Lanasol Brown 2R—(I)	661
Naphtha Chrome Violet 4B—(I)	110

UNIDENTIFIED DIRECT DYES

Name of Dye and Manufacturer	Quantity (pounds)
†Benzo Fast Brown 3GL—(By)	1,662
†Benzo Red 12B—(By)	649
*Chlorantine Fast Brown 3GL—(I)	2,205
†Diamine Catechine B—(C)	100
Diamine Fast Blue FFG—(C)	100
*Diamine Fast Orange EG	47
Diamine Fast Violet FFBN—(C)	200
*Diamine Orange B—(C)	100
Diazo Fast Violet BL—(By)	99

UNIDENTIFIED SULPHUR DYES

Name of Dye and Manufacturer	Quantity (pounds)
Immedial Violet C—(C)	500

UNIDENTIFIED BASIC DYES

Name of Dye and Manufacturer	Quantity (pounds)
Acridine Scarlet J—(DH)	22
Brilliant Acridine Orange 5R—(DH)	110
Flavophosphine GO—(M)	265
Rhodamine 4GD Extra—(A)	112

UNIDENTIFIED, UNCLASSIFIED DYES

Name of Dye and Manufacturer	Quantity (pounds)
Fast Black LB Base Powder—(GrE)	1,120
Paper Fast Bordeaux B—(By)	13

SYNTHETIC AROMATIC CHEMICALS OF COAL-TAR ORIGIN

Name of Chemical	Quantity (pounds)
†Acetophenone	220
†Amyl Salicylate	5.2
†Anisic Aldehyde (Aubepine)	216
*Benzo Eugenol	0.5
*Benzo Iso Eugenol	0.5
†Benzyl Acetate	230
†Benzyl Alcohol	2
†Benzyl Benzoate	222
†Benzyl Butyrate	22
†Benzyl Annamate	5
†Benzyl Propionate	11
†Beta Naphthol Methyl Ether (Yara Yara)	240
*Cetone D	11
†Coumarin	231
*Diphenyl Methane	1
†Ethyl Annamate	2
†Heliotropine	960
*Iso Butyl Benzoate	22
*Indol	1.1
†Methyl Acetophenone	110
†Methyl Anthranilate	777
*Musk Ambrette	685
*Musk Ketone	63
*Musk (Oleo)	2
*Musk Xylene	125
†Neroline	280
*Para Cresyl Acetate	6
†Phenyl Acetaldehyde	266
†Phenyl Acetic Acid	550
†Phenyd Ethyl Acetate	10
†Phenyl Ethyl Alcohol	232
*Phenyl Ethyl Benzoate	6
†Phenyl Ethyl Butyrate	2
†Phenyl Propyl Alcohol	5
†Phenyl Ethyl Propionate	55
†Rose (for soap)	220
†Vanillin	23.6

MEDICINAL, PHOTOGRAPHIC DEVELOPERS AND OTHER COAL-TAR CHEMICALS

Name of Chemical	Quantity (pounds)
*Amidopyrine	250
*Antipyrine	2,120
Aminopyrazolon	2,000
*Attritin	42
†Beta Naphthol (tabloid compound)	6
†Caffeine Sodio Benzoate (Nypoloid)	1
†Cresylic Acid	57,035
†Cresol U. S. P.	1,447
Developer Z	183
†Dianol	19
*Doyen Staphylase (simple)	66
*Doyen Staphylase (Ioduree)	44
*Enesol	7
†Epinine	22
†Hadensa (ointment)	121
*Katanol	4,001
*Newisotropin	62
Naphthol AS-SW	2,240
Naphthol AS-BO	1,120
Naphthol AS-RL	560
†Naphthalene (flakes)	44,019
Ortnoamidophenol (base)	110
†Paraphenylenediamine	2,940
†Paramidophenol	110
Xylidine	1,305
Pilocarpine Salicylate	1
†Resorcine Tech	2,240
*Tercinol	2

CHEMICAL SHOW TICKETS OUT AUG. 20

Tickets for the 1923 Chemical Exposition, which will open on September 17 at the Grand Central Palace, New York, will be sent to exhibitors by the 20th of August so as to permit the mailing of them in plenty of time to reach customers and friends early in September. Exhibitors have been requested to distribute tickets with care so as to cut down the really non-chemical element at this year's Exposition to a minimum. Owing to the crowding at the Exposition in past years during the last three days, the first three days of the week are suggested as the best time to view the exhibits in comfort and avoid the crowds. Likewise, and for the same reason, it is suggested visitors attend the Exposition in the afternoon in preference to the evening.

DU PONT ANNOUNCES SULPHOGENE CUTCH R

E. I. du Pont de Nemours & Co., Inc., announce an addition to their sulphur color line, namely, Sulphogene Cutch R.

This color is very adaptable for combinations in the production of khaki and olive drab shads. Its solubility permits of its application in all types of machines.

NEW HOME FOR VICTOR BUSHMAN & CO.

Announcement has been made by Victor Bushman & Co. to the effect that the offices, laboratory and warehouses of this concern are now located at 110 High Street, Boston, Mass., and that due to larger space and increased facilities this firm is in a position to render excellent service to old and new customers.

FOSTER NOW WITH S. R. DAVID

Charles Foster, formerly New England manager for the Calco Chemical Company, has severed his connection with the above concern, and is now connected with the sales force of S. R. David & Co., Inc.

AMERICAN
DYESTUFF REPORTER

Devoted to the use and application of dyestuffs and the mechanical equipment incidental thereto.

LOUIS A. OLNEY
PROFESSOR OF CHEMISTRY AND DYEING, LOWELL TEXTILE SCHOOL.
Directing Editor

LAURANCE T. CLARK
Managing Editor

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WARREN G. HARDING

IN common with the remainder of the civilized world, we mourn the death of Warren Gamaliel Harding, twenty-ninth President of the United States, gentleman, excellent citizen, humanitarian, and apostle of broad-visioned tolerance. His untimely passing, which so inexpressibly shocked the nation, brought a feeling of actual personal loss to thousands who never even saw him, and marked the end of a worthy, useful life. The fact that his political opponents were ready in their public appreciation of his character and integrity, is eloquent of much. He was faithful to his trust as a public servant; he was earnest and mindful of the country's needs as its chief executive; he was dignified, courteous and kindly—a well-loved man.

His administration saw, among other objects attained, the freeing of the American dye industry from the dual menaces of political strife and foreign threat. He was at heart the friend of the industry and a firm believer in the necessity for its independence. In a letter which he wrote shortly after his election to Dr. Charles H. Herty, then acting as chairman of the Advisory Committee for the Sixth Chemical Show, he said: "The war taught us what we needed to make us truly a self-sustaining, independent nation, and much will depend upon the success of our chemical engineers and producers to remedy the deficiencies which then became more impressibly apparent."

At the spring, 1921, meeting of the American Chemical Society at Rochester, Representative Nicholas Longworth, in an address referring to Senator James W. Wadsworth and himself, quoted President Harding as saying to him: "I hear you and Jim are going to address the American Chemical Society. Well, you can tell the members that what they need more than addresses is protection—and they are going to get it."

This indicates what the attitude of our late President was toward the chemical industries; and while the present tariff law is not, perhaps, a perfect instrument for the achievement of this end, the intent of the law with respect to the dye industry is so plainly stated as to provide it with a most potent weapon of defense in court actions. Thus it may be believed that, so far as he was able, President Harding redeemed his promise to the industry.

For his principles and fairness, then, as well as for his engaging qualities as a man, we join the nation in deep sorrow at his passing.

SOUND ENTERPRISE

THERE is something new under the sun. The sponsors of the Ninth Chemical Show have conceived and will put into effect a plan which, so far as we are informed, will be unique in the annals of trade expositions, and which will make the forthcoming Show more truly worthy of the adjective "educational" than ever before.

The project is nothing less than the offer of a short course in the fundamentals of chemical engineering and industrial chemical practice, consisting of lectures, visits with an instructor to the exhibits, to be followed by a report from the students who take advantage of this opportunity during Show week.

Students of chemistry and chemical engineering were invited to enroll in this course, which will be given by a committee of educators under the chairmanship of Prof. W. T. Read, of Yale University, a member of the advisory committee.

The lectures will be given by men prominent in various phases of the chemical engineering industries, and will be supplemented by group conferences and supervised inspection of the exhibits. A report or examination on certain phases of the course will be required by the committee.

The following topics will be included in the course:

1. Plant Equipment in the Chemical Engineering Industries: (a) Disintegration—Crushing and Grinding; (b) Mechanical Separation—Grading; (c) Separation of Solids from Liquids—Thickening, Filtration, Centrifugal Separation; (d) Separation with Phase Change—Evaporation, Distillation, Drying; (e) Handling of Materials.

2. Materials of Construction: What Material to Use, When, Where and Why.

3. Chemicals in Commerce: The Distribution of Chemicals.

The preliminary announcement of speakers for the above subjects is made as follows: Subjects (a) and (b) will be generally discussed by Galen H. Clevenger, an engineer prominent in this work. Subject (d) will be discussed in general by Prof. W. H. McAdams, of Massachusetts Institute of Technology, whose recent

work and co-authorship of "Fundamentals of Chemical Engineering" created much interest in technical circles. Subject (e) will be discussed by A. E. Marshall, of Baltimore, and other speakers upon the same subject will be Harry Carlson, on "Conveying with Steel Belting"; S. G. Ketterer, on "Pumping by the Use of the Jet Apparatus," and G. L. Montgomery, discussing "Transportation in the Chemical Industry."

Subject 2 will be broadly discussed by W. S. Calcott, chemical engineer of the Du Pont Company, while other speakers will be Ross C. Purdy, secretary of the American Ceramic Society, "Ceramic Materials: Clay, Glass, Silica and Enamelware"; O. I. Chormann, "Glass, Enamel, Steel Products as Materials of Construction," and Homer Hendricks, "Zinc as a Material in Construction."

There will be no charge made to the students attending this course, and already, through their instructors (through whom registration cards may be secured), over 150 students have applied for admission to attend.

Further, in view of this wide interest Columbia University has offered its dormitories to the students attending the course for use during the week of the exposition, making a special rate to these students. Registration cards for the use of students will shortly be distributed through their instructors.

On the score of desirable publicity in connection with the chemical industries and the Exposition—this alone would make the project worth while. But going deeper than that, it reveals the fact that its instigators have firmly grasped the underlying theory of such exhibitions, and have wisely selected a means well calculated to make this purpose more widely discussed and known. What may or may not be actually learned through such a short course is in this case secondary. It reflects credit upon an industry to have produced conditions and habits of thought giving rise to a "stunt" of this nature, and by means of it the existence and role of chemical education is brought prominently into the national consciousness. It will undoubtedly offer to many general students all the advantages of a "trial marriage" to a specific vocation, and who knows but what it may be effective in influencing the final choice in favor of chemistry as a profession in a number of cases!

It is an ingenious and, withal, a sound bit of enterprise, and deserves a thorough trial with the support of the chemical industries to aid it.

SILK ASSOCIATION SENDS CONDOLENCES TO MRS. HARDING

Among the thousands of expressions of sympathy which have been called forth by the death of the late President Harding is a telegram sent Tuesday, August 7, to Mrs. Harding by the Silk Association of America.

"The Silk Association of America," says the message, "joins the whole world in mourning the death of Mr. Harding, who gave of his best at all times. Honest, straightforward, sincere and conscientious, the nation can never fully appreciate the sacrifice he made. Our deepest and most heartfelt sympathy to you in this your hour of grief."

SOCIETY MEETINGS WITH CHEMICAL SHOW

The Chemical Exposition this year is being held during the week immediately following the annual meeting of the American Chemical Society, as has been the custom in previous years. The A. C. S. will meet during the week of September 10 at St. Paul, and immediately after the close of the meeting its members will entrain for New York in order to spend the week of the 17th at the Exposition. The usual meeting of the American Ceramic Society will be held in conjunction with the Exposition in the Grand Central Palace on September 19, probably extending over several days. The annual banquet and election of the Salesmen's Association of the American Chemical Industry will also be held during the week of the Exposition, very probably at one of the Pershing Square group of hotels near the Grand Central Palace. The regular dinner of the American Institute of Chemical Engineers will be on Wednesday evening, September 19, at the Park Avenue Hotel, reservation for which should be sent to Dr. John C. Olsen, secretary, at 85 Livingston Street, Brooklyn, N. Y.

RAW SILK STOCKS 2,000 BALES OFF IN JULY

Raw silk stocks, according to figures released by the Silk Association of America, were smaller by approximately 2,000 bales August 1, than they were the previous month. Imports and deliveries to mills during July, however, were both somewhat larger than in June.

July imports reached a total of 25,622 bales, of which 203 were European, 18,151 Japan, and 7,268 all others. Stocks August 1 amounted to 22,914 bales, composed of 401 European, 16,831 Japan, and 5,682 all others.

Approximate deliveries to American mills during July equaled 28,573 bales, consisting of 502 bales of European, 23,367 of Japan, and 7,704 all others. Stocks July 1, composed of 700 bales of European silk, 19,047 of Japan, and 6,118 of all others, reached a total of 25,865 bales; July imports added to July 1 stocks made a total amount available during the month of 903 bales of European, 37,198 of Japan, and 13,386 of all others, giving a total of 51,487 bales.

TAGLIABUE TO SHOW PRESSURE CONTROLLERS AT CHEMICAL SHOW

The exhibit of the C. J. Tagliabue Manufacturing Company at the Chemical Show will consist of the following representative products:

Tag Perfect pressure controller, a simple, accurate, and rugged controller which automatically maintains a desired pressure with invarying uniformity.

Tag Simplex pressure controller, a smaller brother of the Perfect which maintains pressure within very close limits. The Simplex is especially desirable where

economy of space is a factor as it is mounted directly on the diaphragm motor valve which may be placed either horizontally or vertically.

Tag recording and dial indicating thermometers, incorporating many improved structural features exclusively Tag.

Tag industrial thermometers, featuring in the mercury type the Tag-Hespe red reading column, a broad red line from the top of the tube to the top of the mercury column which, catching the eye instantly, is easily followed *down* to the top of the mercury column where the temperature is read. The exclusive Tag feature, bringing to the accurate mercury thermometer the easy reading qualities of the spirit thermometer.

Tag chemical thermometers and hydrometers.

Tag Mono-Duplex recorder for flue-gas analysis, the only flue-gas recorder that gives a continuous record of *both* CO_2 and combustible gases, a matter of importance in boiler room economy since CO_2 records alone are misleading.

Tag oil-testing instruments, many of which have been adopted as standard by the A. S. T. M., U. S. Government, American Petroleum Institute, etc.

Unshrinkable Finish on Wool

Relationship Between Shrinking and Felting—Common Complaints of Treated Materials—Properties of Chlorinated Wool—Scientific Control

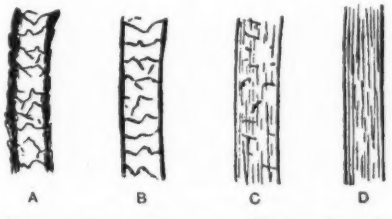
By S. R. TROTMAN, M.A., F.I.C.

Shrinking and Felting Related

THE chief disadvantage of wool, from the point of view of the hosiery manufacturer, is its power of shrinking and felting under the influence of hot soap and water. Both of these properties are very closely related to each other, though not necessarily manifestations of the same phenomenon. Various causes have been suggested to account for these effects. The simplest and oldest theory is that felting is due to the interlocking of the free edges of the epithelial scales. Some chemists hold, however, that it is due to the presence in the fiber of an adhesive gum, while among later theories is that advanced by Justin Mueller, which explains felting as being simply an attribute of the "colloidal" wool substance. According to this theory, wool when boiled with soap and water swells up like other colloids, and then becomes plastic, in which state the fibers, if brought into contact with one another, partly coalesce and permanently adhere to one another. Probably no single theory explains all the phenomena. Thus it is quite certain that any process or treatment which attacks epithelial scales reduces the shrinking and felting power of the wool fiber to an extent roughly proportional to the amount of damage produced. Among chemical reagents which attack the substance of which the epithelium is composed, chlorine or hydrochlorous acid is the most potent, and this fact is the starting point of all the processes employed for producing an unshrinkable finish. For this purpose the scoured goods are treated with a limited quantity of a dilute solution of bleaching powder or sodium hypochlorite in the presence of an acid. The following is an example: The goods are steeped in a solution of hydrochloric acid of a strength of 1.5 to 2 deg. Tw., then squeezed and passed into a cold solution of bleaching powder of about the same density. After working in this for about twenty minutes, they are washed and passed into a bath of sulphurous acid or sodium bisulphite to remove any unused or combined chlorine. They are then washed with water, then in a dilute solution of sodium carbonate to neutralize the acid, and finally with water till free from alkali.

Common Complaints

The process is purely empirical—that is to say, we do not understand what happens, nor much of the conditions under which the reaction takes place. It does not lend itself so readily to scientific control as other



Unshrinkable Finish on Wool.—Fig. 1.

processes—as, for example, bleaching. For these reasons it is hardly surprising that it is somewhat uncertain in its results, and not infrequently damages the fabrics as well as making them unshrinkable. Complaints in connection with unshrinkable goods are generally one of the following: (1) The goods are not sufficiently unshrinkable; (2) though unshrinkable, they break down quickly in wear; (3) much weight is lost during the unshrinkable process. The difficulties of the manufacturer are great. If he does not carry the process far enough he only produces limited unshrink-

ability. On the other hand, if he pushes the action of the bleaching powder to the point where complete unshrinkability is produced, the goods are liable to wear badly, and the loss of weight during the process is very considerable.

Although we cannot explain exactly what happens during the process from a chemical point of view, yet we can follow the course of the reaction by means of a microscope. There is a very important difference between the microscopic appearance of ordinary and chlorinated wool. In the former the free edges of the epithelial scales can always be seen distinctly. In chlorinated wool these are no longer visible; the scales appear to adhere to the cortex throughout. This is seen at A and B in *Fig.* 1. In a well-chlorinated sample the outline of the epithelial scales is still perfectly plain. Their surface is smooth and opaque as in natural wool. Incipient damage is indicated by the outline of the scales becoming difficult to observe, and their surface rough. Sometimes they become so thin that the underlying cortex can be seen as at C. Actual damage is denoted by the complete disappearance of the epithelial scales exposing the cortex of the fiber. This gives the fiber a rough surface marked with longitudinal streaks as seen at D. This damage may be either local or general. It can be estimated quantitatively by counting the percentage of damaged fibers present. Samples of commercial unshrinkable fabrics contain from 5 to over 50 per cent of damaged fibers. Complaints of bad wearing properties are invariably found to be associated with the presence of large numbers of such fibers.

Properties of Chlorinated Wool

The properties of chlorinated wool are generally stated to be: (1) It has a decreased tensile strength and elasticity; (2) it has an increased affinity for dyestuffs; (3) it is more readily wetted down than ordinary wool; (4) it has a scroop, particularly when wet; (5) it has different electrical properties to those of ordinary wool.

I have proved, partly in collaboration with research students of the department, that some of these properties are really those of overchlorinated or damaged wool, while others must be added, such as altered solubility in water, dilute alkaline solutions, perspiration, etc., an increased affinity for atmospheric moisture, and less stability toward the action of air, ozone and micro-organisms.

In general it may be stated that the properties of chlorinated wool differ very little from those of ordinary wool until the stage of incipient damage is reached. From this point onward all the bad properties increase rapidly with the degree of overchlorination. Thus, as soon as the scales begin to disappear there is a marked increase in the solubility of the fiber; it loses its elasticity; it is less resistant to the attacks of common organisms such as bacillus subtilis, and it is much more rapidly attacked by air or ozonized air.

It is obvious that what we want to do is to stop the process just before the critical point is reached, but this is very difficult to accomplish. Many experiments have been carried out, and others are in progress. A brief account of these may be of interest.

Scientific Control

It has been proved that wool is much more quickly damaged by chlorine than by hypochlorous-acid solutiions of the same strength. All the commercial methods have one weak point—namely, that they make use of bleaching powder or sodium hypochlorite under conditions which make it certain that the wool is acted on by nascent chlorine; that is, under the very conditions which should be avoided. As long as the process is carried out by rule-of-thumb methods, this is probably unavoidable. Scientific control has been shown, however, to be quite possible, by those possessed of only a moderate knowledge of chemical technology. But, of course, manufacturers who have to make use of delicate chemical reactions cannot always expect to have a so-called "foolproof" process. Moreover, it has been found also that different types of fiber require rather different treatment—that is to say, the formula must be elastic and suited to the material. This again could be done with a comparatively small knowledge of chemistry and the use of a microscope.

Experiments are now being conducted with the object of putting the reaction upon a quantitative basis and of studying separately the action of each factor which is concerned in the process. We hope in this way to accumulate sufficient experimental data upon which to found a really scientific method of operation. —Textile Manufacturer.

An exhibit showing what chemicals have done in the development of the radio will be one of the educational features of the Chemical Exposition this year. Any exhibitor at the Exposition who manufactures products which in any way enter into the field of wireless telephony will be permitted to place his prodcts in this special radio exhibit. Not only the raw materials furnished by the chemical industry for the radio industry will be shown, but finished articles suitable for use in this field can be entered.

About fifty American manufacturers of fine precision instruments of various types all the way from high-pressure gauges to ultrafine chemical balances will show their goods at this year's Chemical Exposition in New York, according to early estimates. More than an equal number of manufacturers of all kinds of chemical and allied plant equipment such as centrifugals, screens, crushing machinery, pumps, acidproof ware, valves, gears, pipes, drying equipment, filter presses and the like, will show their goods.

The Permutit Process of Water Softening

By G. H. ROME, A.M.I.C.E.

(Read at Nottingham University College Textile Exhibition, July 9)

THE soap-destroying property of hard water may be regarded as the basis of almost all the trouble caused by hard water in textile mills. The initial step in almost every case in the handling of silk, cotton and wool involves the use of soap in boiling off and scouring. In that initial step a hard water will produce effects the results of which will be felt even after the fabric is in the hands of the ultimate wearer.

If soap is dissolved in a water containing lime and magnesium, the lime and magnesium combine with the fatty acids of the soap, forming oleates and stearates of lime and magnesium, which are insoluble and extremely sticky curds. These curds falling on the fibers, yarns or fabrics form "soap spots" which can never be washed off, give a harsh feel to the goods, and are a cause of spotty dyeing and dull finish.

The turning out of uniformly high-class silk goods is a practical impossibility where the water available for the process contains even only a very small degree of hardness. Hard work and constant vigilance will keep the quantity of goods spoiled by uneven dyeing and the other ill effects of a hard water at a minimum, but will never completely eliminate this loss. Hence the silk mill, for the sake of the quality of the product as well as for economy in operation, must have the softest water possible for every step of the process.

Raw silk before winding is subjected to a soaking process in a strong solution of Castile soap and neat's-foot oil to give it the necessary softness and pliability for the winding operation. If the soaking solution is made in hard water, the lime soaps are precipitated in it and mat the fibers, making them exceedingly difficult to wind. Permutized water, absolutely free from lime and magnesium, on the other hand, produces perfect saponification in the soaking solution. The silk yields readily to the soft water and comes from the solution in a well-preserved state of pliability. No lime soap specks can be formed to stick to the fibers and afterwards cause the silk to reharden. Finally, permutized water will materially quicken the soaking process.

In the degumming, or boiling off, soap again enters prominently into the process, with the important difference that boiling off requires greater quantities of soap and water than the soaking. Consequently, all the ill effects of soap dissolved in hard water are intensified in the degumming operation. The danger from soap specks is much greater because the precipitated lime soap sticking to the silk frequently first discolors it, then partially decomposes, and finally often causes the silk to crack.

The saving of soap in the dyehouse using permutized water is considerable. The saving is, of course, in proportion to the hardness of the raw water, but a large silk mill using water of 9 degrees hardness has reported that the soap saving alone paid for the entire installation in less than two years. Any dyer using a water containing even as little as 5 degrees of hardness can readily appreciate this. At a dyehouse using water not quite 4 degrees hard, when the boil-off bath is prepared the lime soaps can be seen to cover the surface of the water in a scum which must be removed as far as practicable by skimming. This takes time, during which the process is at a standstill, but the wages of the men standing idle go on just the same. Wieh permutized water the soap goes quickly into solution, and as there is no lime or magnesium there is no scum and there is no time lost in skimming.

The precipitation of the lime soap on the silk renders the goods noticeably harsh to the touch. Permutized water, on the other hand, leaves the goods extremely soft to the touch. The proper handle and luster is imparted in the processing of piece dye silks only when an absolutely soft water is used.

The extremely harmful effects of the lime soaps which adhere to the silk in spots and streaks are pronouncedly felt in the operation of weighting silk. The sticky lime soap prevents the weighting solution from coming uniformly into contact with the silk. Wherever the fiber is overlaid with the lime soap the tin cannot fasten itself to the silk, which causes the "hungry spots" that make the dyer complain; and rightly, too, because since the tin acts as a mordant or fixitive, the hungry spots will always show up duller than the rest of the fabric and give the unevenly dyed appearance that is the bane of every dyer. This cause of uneven dyeing disappears, of course, in dyehouses using zero-hardness water, which is free from the least trace of lime and magnesium.

Permutit is not a secret compound. There is no mystery whatever about its composition. It is a zeolite—that is, one of a family of chemical substances which can exchange their constituents over and over again. Most textbooks of chemistry give its chemical formula, which is approximately as follows:

Silica	$2SiO_2$
Alumina	Al_2O_3
Sodium oxide	Na_2O
Water	$6H_2O$

In appearance permutit is a granular, porous material resembling ground quartz.

The total hardness of water is made up of temporary hardness and permanent hardness. The temporary hardness is duue to the bicarbonates of calcium and magnesium, which are precipitated as insoluble carbonates when the water is boiled. The permanent hardness is due to the sulphates, nitrates and chlorides of calcium and magnesium, which are unaffected by boiling the water.

When hard water is brought into contact with permutit, the latter instantly exerts its power of base-exchange, abstracting calcium and magnesium from the water and leaving in the water the sodium which it possesses. The softened water is therefore rendered entirely free from calcium and magnesium, and acquires an equivalent quantity of sodium. The sodium salts thus formed in the water are harmless, soluble, and non-destructive of soap, soda and dyestuffs. They prevent the formation of lime soaps or lime lakes in washing and dyeing, and render the water incapable of forming scale or mud in steam boilers or hot-water systems.

The action is a purely chemical one, and no deposit or precipitate is formed during the softening of water by permutit or during regeneration of the permutit by common salt solution.

Indicating permutit for convenience by the symbol Pm, the softening exchanges are represented by the following equations. For the softening of temporary hardness:

$$Na_2 — Pm + CaH_2(CO_3O_2) = Ca — Pm + 2NaHCO_3$$

i. e., sodium permutit + calcium bicorbonate = calcium permutit + sodium bicarbonate

$$Na_2 — Pm + HgH_2(CO_3)_2 — Mg — Pm + 2NaHCO_3$$

i. e., sodium permutit + calcium bicarbonate = calcium nesium permutit + sodium bicarbonate

For the softening of permanent hardness:

$$Na_2 — Pm + CaSO_4 — Ca — Pm + Na_2SO_4$$

i. e., sodium permutit + calciuum sulphate — calcium permutit + sodium sulphate

$$Na_2 — Pm + MgCl_2 — Mg — Pm + 2NaCl$$

i. e., sodium permutit + magnesium chloride — magnesium permutit + sodium chloride

When this process of exchange has gone on for a specified time, all the exchangeable sodium in the permutit will have been replaced by calcium and magnesium from the hard water. At this point the softening capacity of the permutit is exhausted, and it will not soften any more water until regenerated.

The temporarily exhausted permutit is, however, very readily regenerated and restored to full activity. When a solution of common salt of about 10 per cent strength is allowed to simply flow slowly through the exhausted material, the sodium from the salt solution drives out the calcium and magnesium from the permutit, which thus regains its softening power. This to-and-fro process goes on forever without any depreciation of the permutit. The spent salt solution is a clear liquid like water. It can be sent into the ordinary drains, and is entirely unobjectionable.

The regeneration of permutit is indicated by the following equations:

$$Ca — Pm + 2NaCl\ Na_2 = Pm + CaCl_2$$
$$Mg — Pm + 2NaCl\ Na_2 = — Pm + MgCl_2$$

A permutit plant consists simply of a cylindrical tank containing a bed of permutit. Connected with this tank is a small tank in which is prepared the salt solution for regeneration. The control of the plant is easy, and can be carried out by unskilled labor. The attention required varies from twenty to forty minutes per day, according to the size of the installation.

Another type of permutit has been introduced which, in many cases, affords the user great advantages as compared with ordinary permutit.

The new material is named Permutit B. Its principal difference from the ordinary type lies in the time taken for regeneration. Previously a unit was designed to soften for ten to twelve hours continuously. It then required an equal time for regeneration. Where day and night working was the rule, this involved the use of two units.

A Permutit B plant softens for a shorter period, ranging from three to six hours, and is then exhausted. It can, however, be completely regenerated and put to work again in under one hour. The advantages of this are obvious. One unit only is required for day and night working, as the plant is continuously in operation except during the one-hour periods in which it is regenerated. All that is necessary is sufficient soft water storage to provide for one hour's requirements, to enable one unit to be used throughout the twenty-four hours.

Permutit B uses less salt than ordinary permutit, thus effecting a saving in the cost of treatment. It will be seen that the new material brings down the cost of the permutit process considerably by saving one unit where continuous working is required and by reducing the salt consumption.

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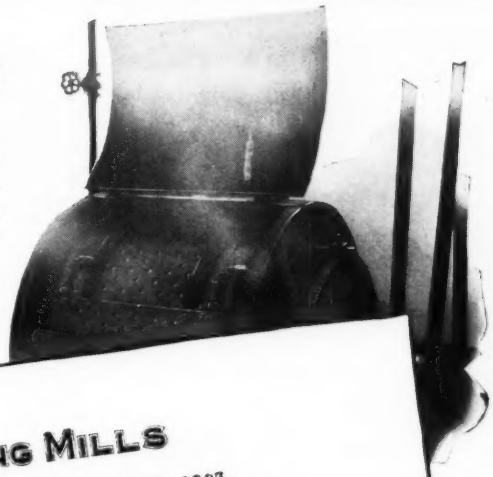

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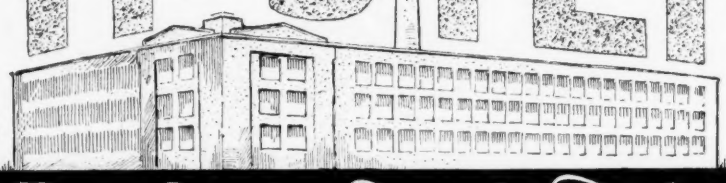

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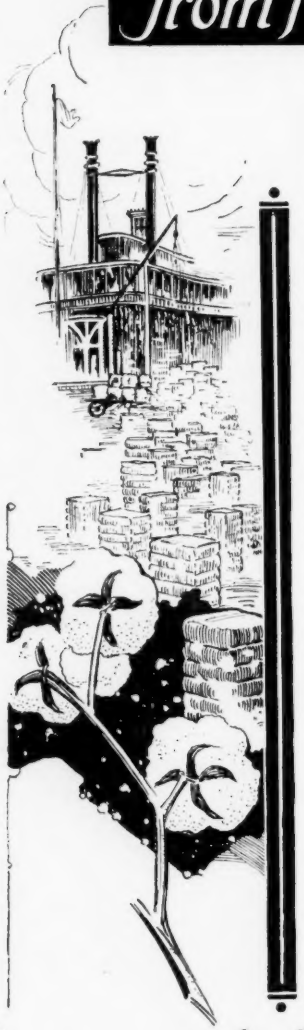

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